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PROCEEDINGS
# of the
SEVENTH
COTTON UTILIZATION RESEARCH
CONFERENCE

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CURRENT SERIAL RECORD

Held at
New Orleans, Louisiana
April 26–28, 1967

Agricultural Research Service
UNITED STATES DEPARTMENT OF AGRICULTURE

PREFACE

The Cotton Utilization Research Conference sponsored annually by the Southern Utilization Research and Development Division brings the results of cotton research (fundamental, chemical modification, and mechanical processing) to the attention of both administrative and research personnel in the textile and allied industries. Special emphasis was placed at the seventh conference on recent developments for the improvement of abrasion properties of durable press cotton products.

This Conference was held April 26-28, 1967, at New Orleans, La. The Honorary Chairman was R. E. Patterson, Dean, College of Agriculture, Texas A&M University, and the General Chairman, Wilmer Smith, of the Plains Cotton Cooperative Association. The program was developed by B. H. Wojcik, Assistant Director, under the direction of C. H. Fisher, Director, in cooperation with staff members, and advisers representing the cotton industry.

These proceedings report in full or in summary the statements presented by the various speakers during the conference and give an account of the discussions following.

Underscored numbers in parentheses refer to references at the end of each paper. The data presented in the references are essentially used as it was supplied by the writer of each paper.

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PROCEEDINGS OF THE SEVENTH COTTON UTILIZATION RESEARCH CONFERENCE

Held at New Orleans, Louisiana, April 26-28, 1967

WELCOME

by

C. H. Fisher, Director
Southern Utilization Research and
Development Division

It is a privilege and pleasure to welcome you to the Seventh Cotton Utilization Research Conference. I am pleased to note that many of our official advisers (names listed on the back of the program) and chairmen of earlier cotton conferences are in the audience. I wish to give them a special welcome and thanks for their valuable help to the Southern Regional Research Laboratory (SRRL).

There are many reasons for believing that this Conference will be a success. We have a splendid audience, a good program, and two Texans as Honorary Chairman and General Chairman. This is a combination that will be hard to beat.

Some good things happened for cotton last year. Most of these are based on or helped by research. One is concerned with the increased consumption of cotton. Mill consumption of cotton increased by 155 million pounds (310,000 bales), or 3.5 percent over 1965 to a total of a little more than 4.6 billion pounds (or 9.2 million bales). The per capita consumption of cotton has increased during each of the past 4 years (from 21.8 pounds in 1963 to 25.1 pounds in 1966 (4, 7)).

The synthetic fibers, however, continued to have their successes. Consumption of all manmade fibers at the mill level in 1966 amounted to about 4 billion pounds, a new alltime record which exceeded the previous high of 1965 by 10 percent (2, 7).

The high per capita consumption of both cotton and the synthetics may be attributed, in part, to research and, in part, to the current U.S. economic expansion, now in its sixth consecutive year. A report from the American Textile Manufacturers' Institute (ATMI) shows that, compared with 1961, the United States in 1966 had 12 million more people; anticipated 1966 consumer expenditures are $117 billion higher; and spending for clothing and shoes is $10 billion greater (1). In 1966 the American public spent $34 to $35 billion on clothing, excluding shoes (10).

It is good news for cotton that the carryover of cotton--recently at the level of more than 16 million bales--will be reduced by about 5 million bales during the 1966-67 year. Most of this reduction in carryover, however, is occurring in the longer staples to the point that there is danger of a real shortage developing for cotton having desirable qualities (5).

This means that an old problem has taken on greater importance. More than 50 percent of cotton in the loan is unattractive for textile purposes because of low micronaire, high micronaire, or short staple length (8, 9). Ralph A. Rusca has studied this situation and has developed a plan of research that appears to have promise of aiding this situation. However, our research facilities are now being used on projects of equal or higher priority.

Good for cotton, also, is the fact that concentrated research on the development of improved durable-press products from cotton is paying off. There are several important advances in this area of research. Papers describing some of these advances will be presented at this Conference.

One approach of making durable-press cotton products is based on vapor-phase treatment. As some of you know, the Gagliardi Research Corporation recently concluded its contract research for the Department of Agriculture on vapor-phase treatment. This work created great interest and caused many inquiries to be directed to the SRRL. To satisfy this need for information, the SRRL sponsored--with the aid of the National Cotton Council--a special conference on the vapor-phase finishing of cotton. This Conference, held on February 24, was unusually successful. About 160 visitors attended the Conference to exchange information on vapor-phase finishing (11).

Promising for cotton, also, has been the establishment of two new research and promotion organizations for cotton. The more recent one will be financed by cotton producers at the rate of $1 per bale. George S. Buck, Jr., of the Cotton Council, will give you more information about this program (3, 6).

A somewhat similar program will be operated by the International Institute for Cotton. The funds for this program, estimated at $6 to $8 million annually will be raised by contributions of $1 per bale of cotton produced by several exporting countries (3).

These two research programs will greatly increase the funds for research and promotion to benefit cotton. Even with this increase, however, cotton will still be at a great disadvantage with the synthetics with respect to volume of research and promotion. It has been reported that about $149 million and $70 million, respectively, are spent annually for research and promotion designed to benefit the synthetic fibers. This means that the synthetics continue to have a great advantage over cotton from the standpoint of research and promotion (3).

Turning now to the development of all-cotton durable-press products, several approaches which were developed in cooperation with the National Cotton Council and other organizations are described below. A few comments will be made first, however, about the total program at SRRL to develop improved durable-press products from cotton.

In September 1964, the Southern Utilization Research and Development Division (SURDD) intensified its research on durable press. The results of this intensified and coordinated effort (about 50 professional man-years per year) have been gratifying. More than 40 research papers and patents describing our work on durable press have been published. Several new methods for improving abrasion resistance of all-cotton durably pressed products have been developed. These include:

(1) Use of improved yarn and fabric structures.

(2) Application of polymers to the surface of the cotton fibers, e.g., selected silicones, urethanes, acrylates, and polyethers.

(3) Mechanical compaction of the fabrics (to be discussed at this Conference).

(4) Preferential crosslinkage, that is, crosslinkage in selected regions of the fabric. (Corduroys are an example.)

(5) Slack mercerization and similar pretreatments. (Example: Slack mercerize and then stretch fabric to about 90 percent of its original width.)

(6) Treatments that swell the fibers and deposit polymers in the fibers before or during crosslinkage (e.g., the Poly-set process).

(7) Use of blends of untreated cotton with resin-treated cotton.

(8) Attachment of long-side chains to the surface of cotton fibers.

(9) Vapor-phase treatments (already mentioned).

The first two advances, listed above, are already being used industrially. The other seven are being evaluated by the textile industry.

It is not yet clear to scientists at SURDD which of these nine individual approaches may be combined most advantageously with others. But, it is known that some can be combined effectively. Presumably, the approach of having improved yarn and fabric structures will apply to most other approaches for improving the abrasion resistance of the finished product. Probably some of the other approaches will be found additive, too.

The new carbamate finishes developed by SURDD scientists are used in the production of about 80 percent of all-white durable-press products.

We feel that much progress has been made in this important area of research. This progress over the broad front, particularly when combined with advances made by other organizations, causes us to have considerable optimism about the future of cotton in durable-press and other products.

Success in research on durable-press cotton products is being reflected in the market place. At least three manufacturers are now offering all-cotton durable-press shirts. Another manufacturer is offering a "fortified" cotton durable-press shirt containing 65 percent cotton. Others are on the horizon.

Some additional instances of noteworthy research and development are:

a. All-cotton, durable-press seersucker suits for men were successfully test-marketed in cooperation with the National Cotton Council.

b. New carbamate finishes were made available commercially.

c. New scientific information on fiber hooks and their processing is being used commercially by the textile industry.

d. The commercialization of improved cotton batting (Cotton Flote) is progressing satisfactorily.

e. The SRRL 720 spindle hour spinning test was useful in the development of a new American Society for Texting Materials (ASTM) test procedure.

f. A new edge abrasion test based on the Accelerotor was developed. This method appears to be as satisfactory--with 1/12 the time--as the conventional laundrying method for evaluating abrasion resistance.

g. The SRRL Bale-Opener-Blender was evaluated by Greenwood Mills. Some of the results, which are promising, were reported by Leland Beaudrot at the last Cotton Research Clinic of the National Cotton Council.

h. Use of textile mill machinery (total of nine) developed at SRRL continues to increase. It is understood that 15 SRRL Opener-Cleaners were installed recently in various mills. About 10,000 Fiber Retrievers and 2,500 Granular Cards are now in use. About 30 mills have used the Loom Attachment.

i. A new flame-retardant tetrakis (hydroxmethyl) phosphonium hydroxide (THPOH), looks particularly attractive for use with light fabrics. More work must be done, however, before we can fully assess the potential of this new treatment. The development of THPOH will be described at this Conference.

j. It is understood that all-cotton molded bras, developed with the aid of SRRL scientists, will be manufactured.

k. Many industrial firms have expressed an interest in the new Zirchrome process for applying (single padding) pearl gray mineral dyes to cotton fabrics. The products have unusually good resistance to rot and weathering.

l. There is strong commercial interest also in the newly developed colorless weather-resistant finishes combining zirconium acetate and methylolmelamine.

m. Additional progress has been made in developing a machine based on electrostatics for separating short fibers (3/8 inch or less) from the longer fibers, and possibly for spinning.

n. A simple mechanical method for imparting filling stretch to fabrics has been developed for the first time. This method, which will complement warp-compaction methods, will be described at this Conference.

Thus far, I have emphasized developments having relatively short-range and practical potential. This should not lead to the erroneous conclusion that long-range basic research is missing from our program. Our program includes a great deal of basic research, which is yielding interesting and profitable information. Recent achievements include:

a. Studies in fiber physics have provided new information about the relationship among properties of fibers, yarns, and fabrics.

b. Microscopy and spectroscopy have been used to obtain new information about fiber structure and the effect of chemical treatments.

c. The effect of swelling and chemical treatments on fiber surface areas have been determined by nitrogen adsorption techniques.

d. Much has been learned about the relative rates with which the hydroxyls of the anhydroglucose units react with various chemicals.

e. Valuable information has been obtained also about the kinetics of cellulose reactions, relation between structure and properties of crosslinking agents (and of the crosslinked cellulose), and the relation between the structure and swelling characteristics of various chemicals.

Other noteworthy events for calendar year 1966 include:

a. Results of research reported in 231 published papers and in many presentations at scientific meetings. About half of these are concerned with cotton research.

b. Research reported in 41 new patents. Additional patent applications (42) were filed, and 27 licenses were granted.

c. The SURDD sponsored, or co-sponsored eight formal conferences attended by 969 registrants exclusive of SURDD personnel.

d. Information disseminated by 53 news releases and 18 radio and television performances. A major proportion of these described advances in cotton research.

e. Wilson A. Reeves, Chief of the Cotton Finishes Laboratory, received the 1966 Olney Medal of the American Association of Textile Chemists and Colorists.

f. Mr. Reeves also received the 1966 Southern Chemist Award.

g. A. M. Altschul, recently appointed Special Assistant to Secretary Orville L. Freeman, received the First Technion Award, conferred by the Chicago Chapter of the American Society for Technion.

h. The Department of Agriculture conferred the Superior Service Award on the group comprising R. L. Acreneaux (deceased), John G. Frick, Jr., Gloria A. Gautreaux, Russell M. H. Kullman, J. David Reid, and Robert M. Reinhardt for their contributions to the development of carbamate finishing agents, valuable in making durable-press products.

i. Wilson A. Reeves named adviser to Assistant Secretary Dorothy Jacobson, U. S. Delegate to the International Institute for Cotton.

j. R. M. Persell received the Honor Scroll Award of the American Institute of Chemists.

We receive advice on our cotton research from a group of official advisers, whose names are listed on the back cover page of the Conference program. We concentrate our research on areas that seem most promising to help cotton. This means that some research areas are not included in our program. Examples of research areas that receive little or no attention are: Nonwoven fabrics, plastic laminates, solvent finishing, blends of cotton with synthetic fibers, dyeing, machinery development for mechanical processing beyond the card, and machinery for wet processing.

Some of you are aware that a new building is being constructed adjacent to the SRRL. This building, which should be ready for use this fall, will house two pilot plants for processing cotton. One of the pilot plants will process cotton mechanically; the other will be used for the chemical or wet processing of cotton. These new facilities will increase our potential--by about 30,000 sq. ft. of floor space--for doing research helpful to cotton and to cottonseed.

In closing, I wish to emphasize that our research and related activities have been greatly aided by cooperation with many individuals and organizations. We are sincerely grateful for this privilege and valuable help. We hope we shall always have this pleasant and profitable working relationship.

I wish to express appreciation to our visitors, chairman, and speakers for their valuable contributions to the Conference.

We plan to hold the Eighth Annual Cotton Utilization Research Conference May 1-3, 1968. We hope to see all of you then.

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OPENING REMARKS

by

R. E. Patterson
Agricultural Experiment Station of the
Texas A&M University
College Station, Tex.

It is indeed a great pleasure to serve as Honorary Chairman of the Seventh Cotton Utilization Research Conference. I am looking forward to the discussions of the research progress being made on cotton and cotton products. This conference will not only provide you in the trade with information about the results of on-going research, but it will also give me an opportunity to get your reaction to the findings.

An administrator of any research program must continually evaluate and reevaluate his program. Research efforts must be directed toward problems of the future, not of the past. The success or failure of a research program depends on the ability to provide answers to questions that will arise tomorrow. Conferences such as this provide invaluable aid in such reevaluation efforts.

The Southern Utilization Research and Development Division has an outstanding record of providing answers to tomorrow's questions. Much publicity has been given discoveries in areas such as durable-press goods, stretch cottons, flame-retardant cottons, all-cotton garments, and other popular uses of cotton. We are pleased with the success in these research areas. But perhaps even more significant are the findings from basic research. Studies on the basic structure of the cotton fiber and the effects of various treatments on its properties will provide the foundation with answers to many future questions.

Frequently, findings from basic research on one problem provide insights into problems of an entirely different nature. For example, last year Mr. Grant reported the results of studies to determine the relationship between the ring structure and strength of cotton fibers. While conducting research on the development of these rings in our controlled environmental laboratory, Dr. Powell, of our Plant Sciences Department, discovered that hot nights were quite detrimental to pollination and fruit set of hand-pollinated flowers of the cotton plant. Thus, investigations into cotton fiber structure provided a lead in understanding the fruit-set process of the cotton plant. We have not yet discovered means to insure cool nights during the fruiting stage, but we have developed cotton plants that will germinate and grow off at soil temperatures below 58° F. This research points the way to cotton that will grow even in early season cool weather with less loss from seedling disease.

Other research at Texas A&M which may interest you is directed toward the development of a longer staple cotton adapted to the Texas High Plains. Still in the experimental stage are several stormproof varieties that will produce fibers from 1-1/32 to 1-1/8 inches in length. These varieties also have excellent fiber strength, but at this point in their development they yield 10 to 15 percent below the best adapted varieties currently being produced in the area. L. L. Ray, of our South Plains Research and Extension Center, near Lubbock, has also grown successfully at that location Acala 1517 and Del Cerro varieties, which have long staple and require a long growing season.

We are well aware of the length-strength, micronaire problem on the Texas High Plains and hope to develop improved varieties that will allow this area to produce better cotton. Such varieties with improved quality must presently sell 2 to 4 cents above the price of the most commonly used higher producing varieties for producers to break even and compensate for their reduced yields. The 1967 loan price differentials will give the producers an added incentive to improve their quality.

I am very glad Wilmer Smith is here as Chairman of this Conference. He is well aware of these problems and is instrumental in bringing about quality improvement in the area. I am confident that you will see some major changes in High Plains cotton quality within the next few years.

We are still badly in need of research on efficient uses of low-quality cotton. The Cotton Research Advisory Committee strongly urged this type of research in their meeting last March. They indicated the highest priority should be given to "Expanded research to improve and find new uses for short-staple cotton."

A recently proposed research program on low-grade cotton has been prepared by Ralph A. Rusca, Chief of the Cotton Mechanical Laboratory at the Southern Utilization Research and Development Division. We enthusiastically support such research. Earlier, I mentioned prospects for improvement in cotton quality on the Texas High Plains. We realize we have a long way to go.

In 1966, the High Plains of Texas produced an estimated 1,310,000 bales of cotton. This is more cotton than produced in any state other than Texas and Mississippi in 1966. More than one-half of this cotton had a micronaire of 3.2 and below--a micronaire considered unusable except for a few low-grade items. About 66 percent of the 1966 crop from the Texas High Plains was 15/16 inch in length, or less--almost 87 percent of the cotton was shorter than 1 inch. Also, about 88 percent of this cotton had a Pressley Strength below 80,000 pounds per square inch. From these few statistics you can get some idea of the problem we face on the High Plains. Even though last year was an unusually short season throughout most of the High Plains, early freezes occur frequently enough to cause a major quality problem. Of course, Texas is not the only producer of short-staple, low-mike, weak cottons, but by all odds, we are the biggest source of low-grade cotton.

Research on new and efficient uses of this cotton such as proposed by Mr. Rusca may help provide answers to some of our problems. In addition to developing new uses and blends for low-grade cotton, careful consideration of the economics involved in such uses is required. Potential users of low-grade cotton must know what it will cost them and what it will save them before they can be expected to take much interest in the findings. I would like to suggest that a complete economic evaluation be applied to the results of any new findings in uses for low-grade cotton. If this cotton cannot be marketed and efficiently used at its present price, we need to know at what price it can be marketed. Perhaps we may look forward to a report on such research at a future Cotton Utilization Research Conference. For the next 2 days, however, we can look for some interesting discussions of research already in progress.

FIRST SESSION: F. B. Shippee, Research and Development Corporation,
Phillips-Van Heusen, Chairman

STUDIES ON PROPIONYLATION OF COTTON CELLULOSE FIBER

[Summary]

by

A. K. Sircar and D. J. Stanonis
Plant Fibers Pioneering Research Laboratory
Southern Utilization Research and Development Division

(Presented by A. K. Sircar)

Heterogeneous propionylation of cotton cellulose fiber in the form of yarn was carried out with propionyl chloride in a medium of pyridine and dimethylformamide (DMF).

The studies may conveniently be divided into three parts: (1) An explanation of the apparent anomaly between the degree of substitution (d. s.) as indicated by saponification and by weight gain; (2) the supramolecular structure of the propionylated cotton cellulose; and (3) their mechanical, thermal, and thermomechanical properties.

It was observed that the d. s., based on weight gain of the yarns, was much higher than that based on ester saponification measurements. The gas-liquid chromatograms of the pyrolysis products of all the samples showed, in addition to the propionate peak, an extra peak. This was eventually identified as due to 3-pentanone which could arise from the pyrolysis of α-propiolylpropionate, a substance already proposed by Malm and coworkers as a possible side product, but not investigated by them. This compound was further confirmed by the infrared and NMR spectra of the liquid collected on pyrolysis of the sample. Quantitative experiments with the gas-liquid chromatograph revealed the composition of the samples and showed that one out of every four hydroxyl groups is substituted with an α-propionylpropionyl moiety, the rest with simple propionyl groups. A possible mechanism for the side reaction has also been proposed.

The supramolecular structure of the esters was studied by kinetic analysis and by measurement of density, refractive index and X-ray diffraction.

The Sakurada type plot of d. s. against time of reaction showed a discontinuity at a d. s. of about 2.0, where the X-ray diffraction pattern shows almost complete loss of crystalline structure. The assumption that the Sakurada curve actually represents the sum of two simultaneous rate processes, acting in the amorphous phase and at the crystallite surfaces, seems to explain the kinetics adequately. The fiber density decreased continuously, consistent with the idea of continuous destruction of crystalline structure with progressive substitution. Refractive indices of the cellulose decreased continuously with substitution and birefringence, strong in the beginning, was almost absent at complete substitution. The calculated value of molar refraction for the sample of d. s. 2.94 agreed closely with the experimentally observed value. X-ray diffraction intensity traces gave convincing evidence of the progressive decrystallization of cellulose with increase in the degree of substitution. Decrystallization seems to be more or less complete at about a d. s. of 2.0.

Measurements of mechanical, thermal, and thermomechanical properties of the propionylated cotton yarn of varying d. s. included yarn number, breaking strength, elongation, yarn twist, tensile stiffness, differential thermal analysis (DTA), thermogravimetric analysis (TGA) as well as stiffness, elastic recovery, and work recovery at a range of temperatures to a maximum of 155° C.

Breaking strength, on the basis of the tex of the control yarn, is retained within 84 percent of the control up to a d. s. of 2.0. Elongation-at-break generally increases and tensile stiffness decreases with increasing d. s. A great improvement of tensile toughness is observed with increasing propionylation, except in those cases where the sample was evidently degraded by a two-stage preparatory treatment. DTA tracings show exothermic transitions consistent with the idea of recombination and interreaction of pyrolysis fragments of the cellulose molecule and these interpretations are amply supported by the TGA tracings. Stiffness, work recovery, and elastic recovery measured within the temperature range of 30° C. to 155° C. all show second order transitions at about 120 ± 10° C. The rate of decrease of the stiffness values increases with increasing substitution. Also, the steepness of the elastic recovery and work recovery curves at the point of the minimum increases with increasing substitution, consistent with the greater amorphous content. In all the measurements, the curves in the second cycle of heating and cooling reveal the same features as in the first but they are more symmetrical owing to the removal of moisture and doubtless to stabilization of the yarn structure.

DISCUSSION

Question: I am surprised at the disappearance of all crystalline structure in the propionylated cotton yarns, which is in contrast to the usual experience with acetylated cellulose. Can you offer an explanation?

A. K. Sircar: The loss of crystallinity with progressive increase in degree of substitution and the failure to recrystallize upon heating the yarns are believed to be due to the greater bulkiness of the propionyl groups as compared to acetyl and to the presence of about 25 percent of propionyl propionate groups which are probably randomly distributed and would prevent the orderly rearrangement of the cellulose chains into a new crystal lattice.

CERTAIN FINE STRUCTURE AND THERMAL PROPERTIES OF BENZHYDRYLATED COTTON CELLULOSE

by
D. J. Stanonis, W. D. King, P. Harbrink, and C. M. Conrad
Plant Fibers Pioneering Research Laboratory
Southern Utilization Research and Development Division

(Presented by D. J. Stanonis)

SYNOPSIS

The results of a preliminary study of certain physical and thermal properties of benzhydrylated cotton yarns will be presented. The 200-yard lengths of 7/2 yarn consisting of purified mercerized cellulose were reacted in a special reactor to various degrees of substitution (d. s.) from d. s. = 0. 31 to 1. 22 with benzhydryl bromide in a mixture of equal volumes of 2, 6-lutidine and dimethylformamide. The products were examined for tensile strength at break, which decreased only slightly with substitution; ultimate elongation, which was relatively uniform at 60 to 70 percent of the control yarn; and tenacity, which decreased with substitution largely due to added weight. The energy of rupture remained relatively constant at about 50 percent above the control. Tensile stiffness decreased progressively with substitution to about 25 percent of the control. Density decreased 10 to 11 percent with substitution. Crystallinity of the cellulose largely disappeared with substitutions above d. s. = 1, and the lattice of benzyhydryl cellulose began to appear. Stiffness, elastic recovery, and work recovery were measured on 5-inch specimens of the yarn in an oven while they were subjected to repeated extension to about 1 to 2 percent and relaxed as the temperature was raised twice to 200° C. and lowered. During the heating phase of the first cycle the yarn underwent considerable heat adaptation. However, during the cooling phase and during both heating and cooling phases of the second cycle the yarns showed generally favorable behavior. Attempts are made to interpret the results in terms of molecular modifications.

INTRODUCTION

Benzhydryl cellulose is a relatively new member of the cellulose ether family. It was first prepared (6) in 1960 by reacting cotton cellulose with benzhydryl (diphenylmethyl) bromide in 2, 6-lutidine. Although cotton yarns were modified in that work, the structural and mechanical properties of the benzhydrylated yarns were not discussed.

More recently (3) an improved method, employing dimethylformamide as a diluent, was used in the production of benzhydrylated cotton. The prepared yarns were examined by differential thermal analysis (DTA) and by thermogravimetric analysis (TGA) (3).

The same yarns used in the DTA and TGA studies have been examined for certain changes in mechanical and structural properties caused by progressive benzhydrylation. The results are presented.

MATERIALS AND METHODS

The yarn samples investigated in this study have already been briefly described (3). The treated yarns had degrees of substitution ranging from d. s. = 0. 31 to 1. 22 with respect to benzhydryl groups. Yarn properties are reported for the untreated original 7/2 commercially mercerized cotton yarn; and a portion of it that had been put through the entire process with all reagents present except the benzhydryl bromide.

Chemical modification of the yarns. -- Because the detailed conditions of preparation may be more important in a study of structural and mechanical properties than in the previous study (3), some additional information is furnished.

Forty grams of commercially mercerized, nominally 7/2 (167 tex) cotton yarn (0.25 mole cellulose), wrapped on the yarn holder of the reactor described by Stanonis and coworkers (7) was activated with 17.5 percent sodium hydroxide solution. The yarn, still on the holder, was then washed successively with water, 5-percent acetic acid, water, pyridine, and finally lutidine. To the lutidine-washed yarn was added a solution made by mixing 184 ml. of 2,6-lutidine, 184 ml. of N,N-dimethylformamide and 317 grams (1.2 moles) of benzhydryl bromide. The reactions were carried out in the reactor for various lengths of time at 120° C. At the end of the preselected period, the reaction was terminated and the samples washed successively with pyridine, methanol, and water. The results are given in table 1. The kinetics of the reaction approximated that expected on the basis of a Sakurada plot (2).

The test methods have been previously described (2) except for certain modifications that were introduced in a few cases.

The X-ray diffractograms were prepared as described before (1) except that instead of using the tracings, the number of seconds required for a count of 2,000 was automatically printed out for each 0.25 degrees 2Θ over the range 2Θ = 5 to 30 degrees and the reciprocal used as a measure of intensity. The precision in terms of standard deviation of any point on the curves was, therefore, equal to about 2.2 percent. Crystallinity was also determined by the Wakelin correlation method (8) by use of a computer program for the range 2Θ = 10 to 30 degrees.

Tensile stiffness and elastic and work recovery were determined simultaneously at temperatures from 25° to 200° C. on 5-inch specimens in an insulated oven mounted on the Instron crosshead (2). Previous studies (3) showed that no detectable degradation occurred at 225° C.

The overall dimensional change in the stainless steel rods on which the specimens were mounted was determined for the same temperature range by means of 5-in. lengths of copper wire and quartz thread of known linear expansion. Corrections were determined and subtracted from the observed extensions in all cases. During a series of thermal observations, a stream of nitrogen was bubbled through a standard saturated salt mixture (NH_4Cl and KNO_3 mixture giving 69 to 71 percent RH at room temperature), and then through the oven at a rate of 100 to 125 cm^3/min. The crosshead of the Instron was set to move at a rate of 0.2 in./min. both up and down. Readings of temperature, maximum load, and integrator areas were made at intervals of 5 min. as the temperature was raised at a rate of approximately 2°/min.

Table 1.--Conditions and details of yarn preparation

Type of sample	Medium used	Length of treatment	Yarn number	Degree of substitution
		Min.	tex	
Original	None	0	200.2	0
Control	Lutidine, N,N-dimethylformamide	80	197.0	0
Reacted	Lutidine, N,N-dimethylformamide, benzhydryl bromide	20	231.6	.31
Reacted	Lutidine, N,N-dimethylformamide, benzhydryl bromide	45	268.4	.46
Reacted	Lutidine, N,N-dimethylformamide, benzhydryl bromide	90	378.6	.87
Reacted	Lutidine, N,N-dimethylformamide, benzhydryl bromide	210	418.8	1.12
Reacted	Lutidine, N,N-dimethylformamide, benzhydryl bromide	150	391.0	1.13
Reacted	Lutidine, N,N-dimethylformamide, benzhydryl bromide	160	431.0	1.22

RESULTS

Tensile properties. --The tensile properties of the different samples are recorded in table 2, according to degree of substitution. It will be noted that the breaking strength remained relatively constant, but decreased somewhat at substitutions above d. s. = 1. Elongation-at-break increased to nearly double the untreated control (to 10 to 11 percent) except for the treated control for which it increased nearly threefold. To what extent this increase is due to contraction in the process is not known. The small change in yarn number (table 1) speaks for very little contraction, but the uniformity of the yarn itself may not be high enough to justify this conclusion.

Tenacity, reflecting as it does the weight increase owing to combined substituent, decreased regularly to about 40 percent of the control at a substitution of d. s. = 1. The energy of rupture (area under the tenacity--elongation curve) being highly dependent on the elongation is greatly increased for the treated control and remained relatively constant at about 50 percent above the control with increasing substitution. Tensile stiffness shows a somewhat irregular decrease with substitution to a minimum of about 25 percent of the control at substitution above d. s. = 1. In general, the yarns were all of textile quality.

Density. --The density of the cellulose and cellulose derivatives of the yarns is presented in table 2 and is plotted against d. s. in figure 1. It will be observed that the density first decreases rapidly with increasing substitution, eventually reaching an approximately constant value 11 percent below its initial value.

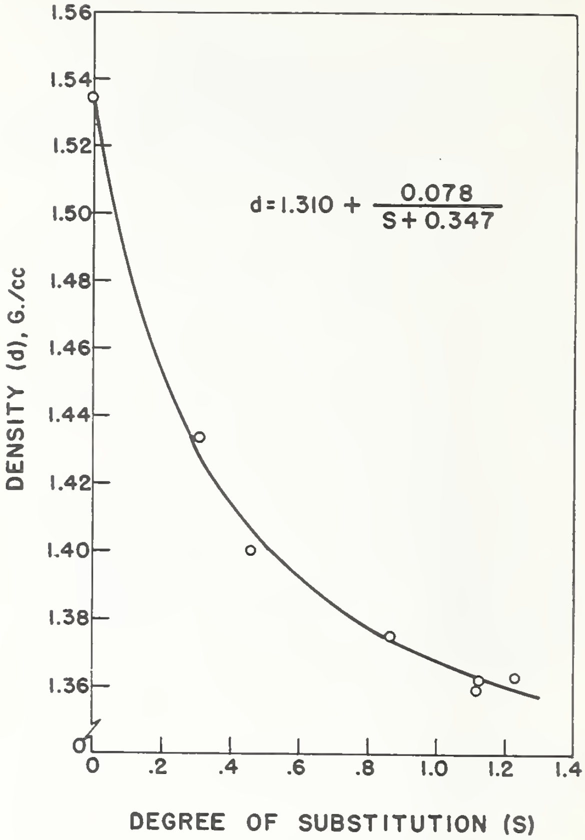

Figure 1.--Change of density of cotton cellulose with degree of benzhydryl substitution.

Table 2. --Tensile properties of benzhydrylated yarns

DS	Breaking strength[1]	Elongation	Tenacity	Energy of rupture	Tensile stiffness	Density
	g. /tex	Percent	g. /tex	g. /tex	g. /tex	g. /cc.
0 [2]	25. 1	6. 3	25. 1	0. 79	397	1. 536
0 (C)[3]	26. 4	18. 6	26. 4	2. 45	142	1. 531
0. 31	25. 3	10. 5	21. 5	1. 33	205	1. 433
0. 46	27. 1	11. 8	19. 9	1. 59	169	1. 400
0. 87	24. 8	10. 7	12. 9	1. 33	120	1. 375
1. 12	21. 6	11. 5	10. 2	1. 25	88	1. 359
1. 13	21. 5	11. 3	10. 8	1. 21	96	1. 362
1. 22	22. 8	10. 9	10. 4	1. 24	96	1. 363

[1] Breaking strength, based on tex of control.
[2] Untreated original, commercially mercerized yarn.
[3] Control treated for 80 min. at 120° C. with all reagents except benzhydryl bromide.

Crystallinity.--The crystallinity of the cellulose of the different samples is indicated by the diffractograms of figure 2. It will be observed that the diffractogram of the untreated control (curve A) is that of relatively crystalline mercerized cellulose, but that it still shows evidence of the native cellulose pattern at 2Θ = 14.7°, 16.3°, and 22.6°. By the Wakelin correlation method (8), this sample had an estimated degree of crystallinity of 65 percent. However, before reaction with benzhydryl bromide it was further reacted with 17.5 percent sodium hydroxide which would have removed most of the residual native cellulose pattern. After exposure to the reaction media and temperature for 80 min., the crystallinity of the treated control was estimated to be 70 percent.

The introduction of a small number of benzhydryl groups (curve B) occurs largely at the expense of the native crystalline component, leaving a sharp hydrate crystal pattern. Further introduction of benzhydryl groups (curves C and D) takes place at the hydrate crystalline regions, gradually reducing them to an essentially amorphous state (curves F and G). The peaks of the latter at 7 to 8° 2Θ are due to subsequent ordering of the benzhydryl cellulose formed.

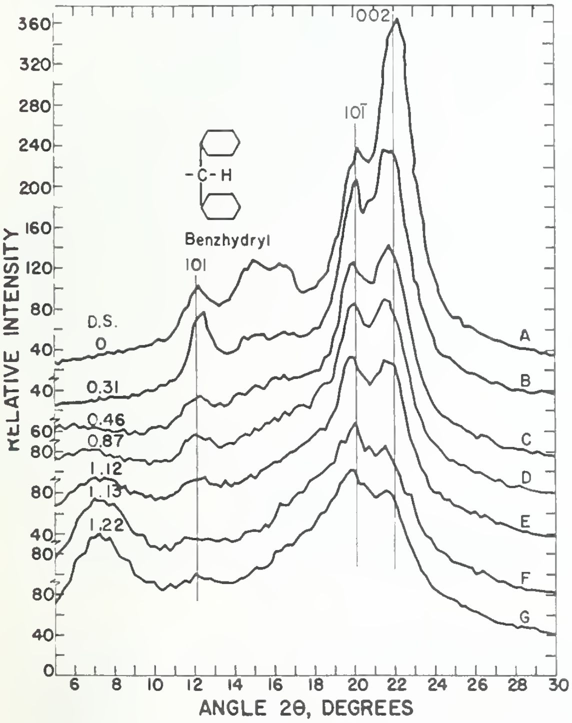

Figure 2.–Diffractograms of benzhydrylated cotton cellulose of different degrees of substitution.

After four of the 5-in. specimens of the more highly substituted (d. s. = 1.12 to 1.22) yarns had been subjected to two cycles of heating at 220° C. and cooling to room temperature in the tensile studies, they were cut and examined by X-ray diffraction. Comparison of the resulting tracings with the curves before heat treatment showed that any changes in X-ray pattern were negligible. Thus, no additional crystallization occurred (2).

Stiffness.--The tensile stiffness of benzhydrylated yarns of different degrees of substitution are shown in figure 3. Each yarn specimen was carried through two cycles of heating to 200° C. or slightly above and cooling. The changes of stiffness with temperature during the heating phase of the first cycle are indicated by dashed curves in the left-hand portion of the chart and identified by d. s. numbers. It will be observed that in almost all cases the stiffness values, as

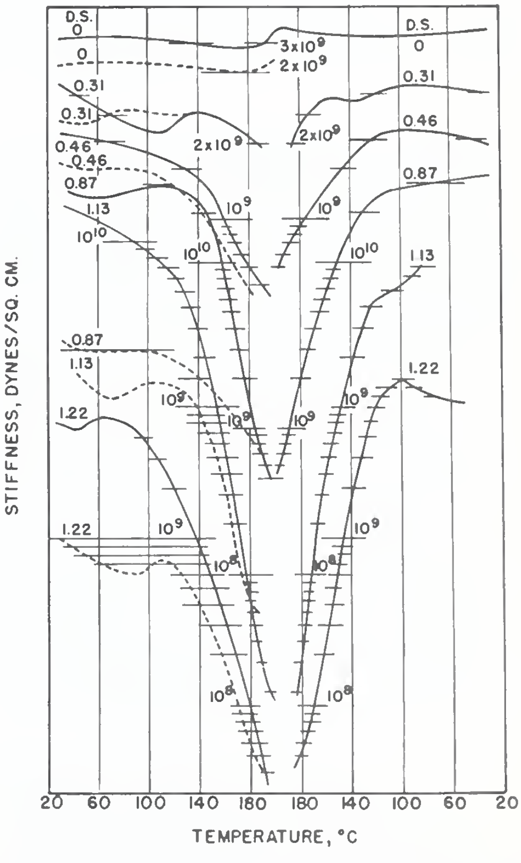

Figure 3.–Stiffness of cotton yarns of different degrees of substitution at different temperatures and cycles of treatment: - - - - First cycle; ——— second cycle.

indicated in dynes per square centimeter by the grid lines and numbers, are considerably below those of the corresponding smooth curves representing the heating phase of the second cycle. Thus, an important change in tensile stiffness is imparted to the yarn by the stressing and relaxation during the heating phase of the first cycle.

During the cooling phase of the first cycle, the yarn displays a tensile stiffness which is almost identical with that followed during both the heating and cooling phases of the second cycle. In fact, the tensile stiffness curves during the cooling phases of the first and second cycle were so nearly identical that they could be represented by single stiffness curves, and no dashed curves are shown. As is evident, the tensile stiffness undergoes a very large change with temperature, in some cases approximately three decades. Owing to beginning thermoplastic flow in samples with the higher substitutions, it was not possible to work at temperatures much above 200° C., although in a few cases temperatures were raised to 220°. As pointed out above, under discussion of the X-ray diffractograms, there was little, if any, change in the X-ray pattern as a result of heat treatment.

Elastic Recovery. --The elastic recovery from deformation at different substitutions and temperatures is shown in figures 4 and 5. Figure 4 shows the thermal behavior during the first cycle of heating and cooling. During the heating phase of the cycle, it is evident that, with increasing temperature, recovery is generally decreased below that of the control even at the lowest degree of substitution (d. s. = 0.31) and that the loss becomes quite large at substitutions of DS = 0.87 and above. There appears to be a minimum in the recovery curves at 120° to 180° C., indicative of second-order transition behavior. However, the elastic recovery fails to show these minima during the cooling phase of the cycle where recovery is rapid. The very large elastic recovery at temperatures below 120° C., especially for the three higher substitutions, is especially noteworthy.

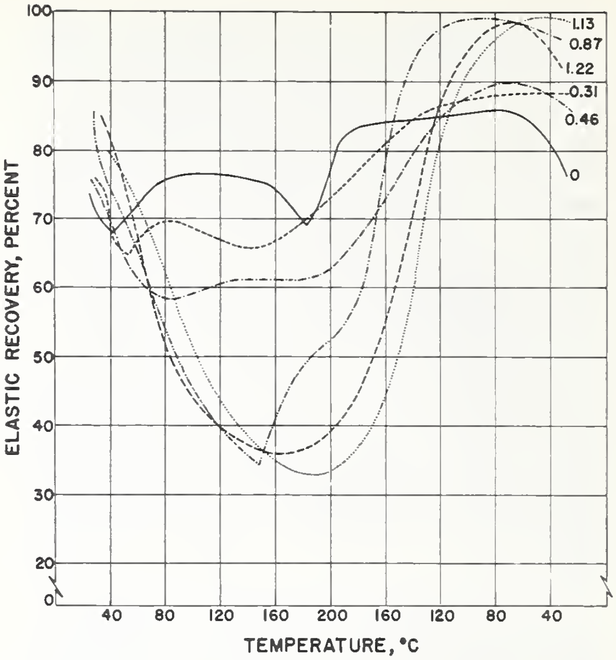

Figure 4.--Elastic recovery of benzhydrylated cotton yarns of different degrees of substitution during the first cycle of heating and cooling.

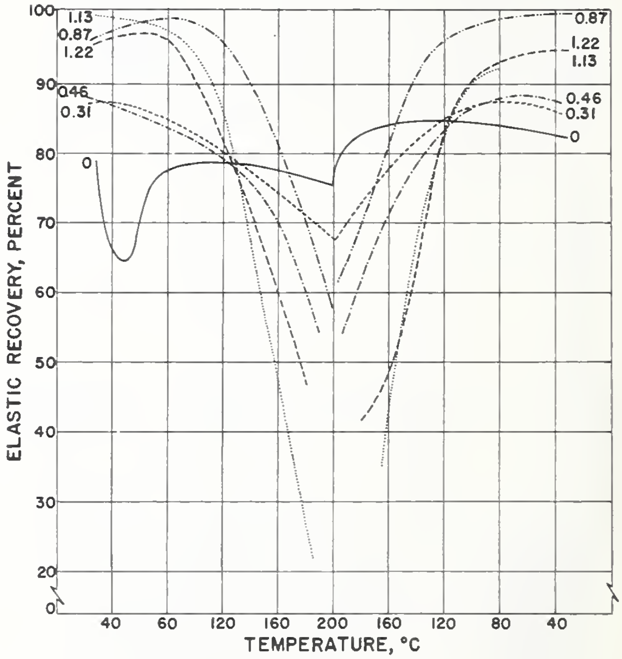

Figure 5.--Elastic recovery of benzhydrylated cotton yarns of different degrees of substitution during second cycle of heating and cooling.

Figure 5 shows the change of elastic recovery with temperature during the heating and cooling phases of the second cycle. It will be noted that, with the exception of the control sample, the curves are quite symmetrical with respect to temperature. They essentially duplicate those of the cooling phase of the first cycle. Also, it will be noted that all substituted samples, especially the three most highly substituted ones at temperatures of 120° C. or lower, have considerably improved elastic recoveries as compared with that of the control sample. Above 120° C. the recoveries are inferior to that of the control sample. No minima, indicative of second-order transitions, are to be seen.

Work Recovery. --The work recovery at different substitutions and temperatures is shown for the first and second cycles of heating and cooling in figures 6 and 7, respectively. Figure 6 for the first cycle shows relatively low recoveries during the heating phase and that recovery is less than the control at all temperatures above 80° C. There are very distinct evidences of second-order transition phenomena at about 140° C. This is further supported by several of the curves (see d. s. = 1. 13 and 1. 22) during the cooling phase of this cycle. During cooling the work recovery at temperatures below 120° C. is improved relative to the control, especially at substitutions of d. s. = 0. 87 and above.

The work recovery curves for the second heating and cooling cycle (figure 7) are quite symmetrical and similar to those for the cooling phase of the first cycle. Now, at temperatures below 120° C. curves for all substitutions show superior recovery as compared with the control. Well-characterized, second-order transition phenomena are indicated. It appears that an important limit occurs between d. s. = 0. 46 and 0. 87 at which the degree of substitution contributes to optimum recovery behavior.

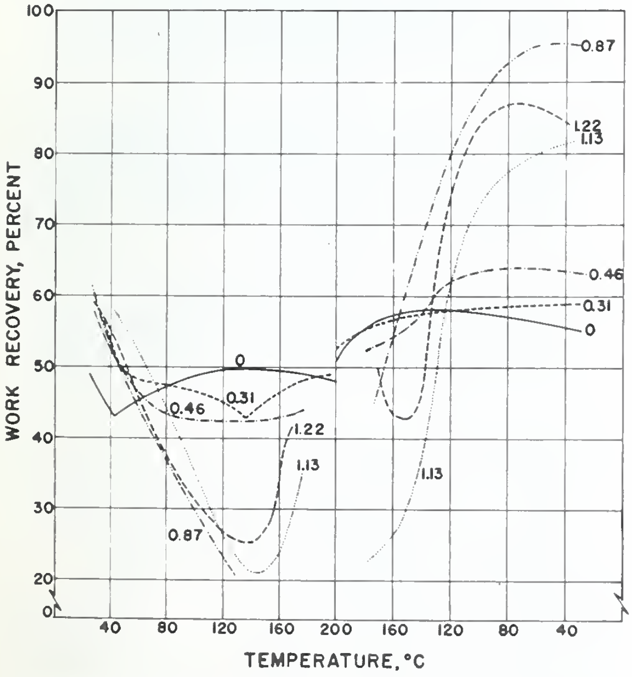

Figure 6.—Work recovery of benzhydrylated cotton yarns of different degrees of substitution during the first cycle of heating and cooling.

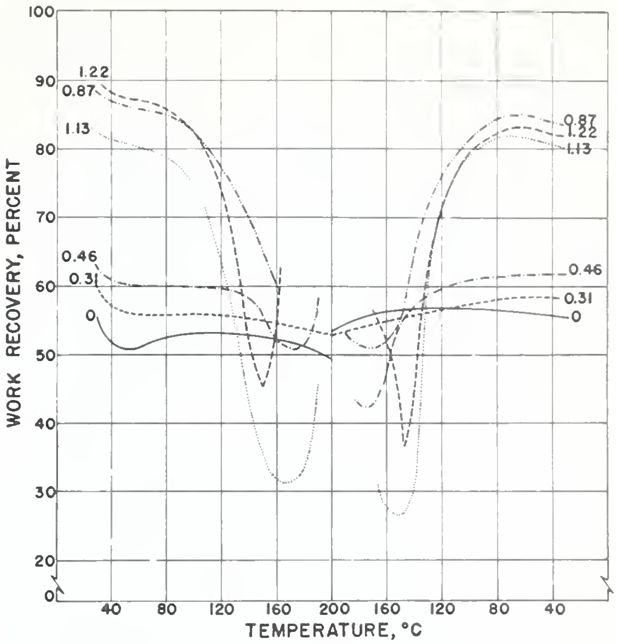

Figure 7.—Work recovery of benzhydrylated cotton yarns of different degrees of substitution during the second cycle of heating and cooling.

DISCUSSION

The present study shows that the cellulose in cotton yarns can be benzhydrylated to about 40 percent of its total hydroxyl groups without loss of textile properties and with a number of interesting changes in physical properties.

Relatively little of the original breaking strength is lost. Thus, it appears that side-bonding contributes in a relatively minor way toward the original strength. If an appreciable proportion of the original strength were due to side bonds, it would be expected that the introduction of the bulky benzhydryl groups would result in a much greater loss of strength as substitution increases. Energy of rupture, on the basis of the untreated control is considerably increased at all substitutions while tensile stiffness decreased noticeably (76 percent).

The decrease in density (11 percent) is associated with the following changes in the crystal lattice:

	a	b	c	β
Cellulose II	8.14A	10.3A	9.14A	62°
Benzhydryl cellulose	12.90	10.3	12.40	41

From the increase in unit cell dimensions a 60 percent increase in volume can be computed. However, on the basis of four anhydroglucose units included in the volume and a molecular weight of 167 for each benzhydryl group a much larger percentage increase would be computed for the weight at d. s. = 1.22. This would lead to an increase of density rather than a decrease. Presumably, the decrease is associated with the noncrystalline components of the cellulose. It appears from this, as well as from the fact that the X-ray diffractograms show much loss of crystallinity that a good deal of loosening of the cellulose structure occurs. Some of this may have been brought about by the alkaline reactivation step in the process but much of it results, no doubt, from the introduction of the bulky diphenylmethyl groups into the cellulose chains.

The results of the present study resemble in a number of ways those of previous studies (2, 4). The density invariably decreases and the stiffness usually decreases. Also, during mechanical working at elevated temperatures the stiffness and elastic and work recovery curves during the heating phase of the first cycle differ quite impressively from those of the cooling phase and both heating and cooling phases of the second cycle. Presumably, considerable movement and accommodation of the cellulose molecules occurs during the first phase of this process.

Unlike the results of earlier studies (1, 2, 5), there was little crystallization of the disorganized cellulose. This should have been indicated by the X-ray diffractograms made on the 5-in. specimens remaining from the thermal studies and probably would have shown up in the DTA curves (3). This failure may again be associated with the bulky nature of the benzhydryl groups which in the cellulose chain cannot accommodate themselves appreciably to the requirements of the crystalline lattice. This failure may have an element of benefit in it in contributing to the favorable thoughness or energy to rupture.

ACKNOWLEDGMENTS

The authors thank the Textile Testing Investigations for physical tests on the yarns and Verne W. Tripp for the diffractograms and for computing the lattice dimensions of the reacted samples.

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THE MICROPOROUS STRUCTURE OF MODIFIED COTTONS

[Summary]

by
B. R. Porter and S. P. Schreiber
Cotton Physical Properties Laboratory
Southern Utilization Research and Development Division

(Presented by B. R. Porter)

Gas adsorption techniques are commonly used to measure the total area and the size distribution of the pores in solid catalysts. More recently these techniques have been extensively employed in the interpretation of the microstructure of molecular sieve materials. Since these measurements are measurements of "reaction sites or areas" and evaluate areas available for reaction and description of the internal morphology of the catalytic materials, it was felt that the method could be useful for similar evaluations of internal structures of cotton fibers. The micropore area of the swollen or unswollen, unmodified fiber gives a measure of the sites available for reaction; the area of the chemically modified product, a measure of the action of the modifying agent on the fibrillar arrays that form the pores within the fiber. In addition, analyses of the complete adsorption-desorption isotherms may yield information on the shape and size distribution of the pores, or voids, within the fiber and the changes in shape and size distribution pursuant to swelling or chemical modification. Such studies, along with corollary studies by methods of electron microscopy, may aid in interpretation of changes in fiber morphology, and help to direct the textile chemist in development of improved cotton products. Only a few such studies have been reported thus far on cottons, but a more extensive literature exists on the application of gas adsorption studies of wood products.

Recent developments of dynamic methods of measurement of gas adsorption isotherms have made this technique feasible for more extensive application to cottons in the developmental laboratory. The studies reported in this paper were made using a dual channel (a sample channel and a reference channel) gas-flow type of adsorption instrument. Samples, in the fabric form, were prepared for testing by swelling in water and exchanging through dried methanol and n-pentane to helium. A mixture of approximately 10 percent nitrogen in helium is used for adsorption, and the sample tubes are immersed in liquid nitrogen during the tests.

As the pressure is increased stepwise, an adsorption isotherm is developed which, up to relative pressures of approximately 0. 3, is assumed to represent the formation of a monolayer of liquid nitrogen. The equations of Brunauer, Emmett, and Taylor when used to evaluate data up to this point on the isotherm give a measure of the total amount of nitrogen adsorbed. Assuming the area occupied by a nitrogen molecule to be 16. 2 angstroms, the area covered by a monolayer of the gas may be calculated. When the pressure has been increased to approximately 0. 96, it is then decreased stepwise to form a desorption isotherm. In all the cottons studied thus far desorption has displayed hysteresis, the amount of which has shown considerable variation. Based on the model of the pore as a closed-end cylinder, the methods of Cranston and Inkley have been used to analyze the complete isotherms and have shown differences in pore size distribution among some cottons. In others, the analysis showed the model assumed was not correct.

The surface areas have been determined of approximately 20 samples of cotton fabrics modified to give improved crease recovery properties. The agents used to modify the cottons included: (1) Formaldehyde applied by a pad-dry-cure process, in the vapor phase and by processes in which the cottons were crosslinked while swollen with different amounts of water; (2) a melamine formaldehyde condensate, applied in several concentrations at different values of pH and cured both wet and dry, at high and low temperatures; (3) butadiene diepoxide applied form media containing sodium hydroxide or methanol; (4) tris-(1-aziridinyl)phosphine oxide. Only two samples retained more than 50 percent of their original area. These were samples treated by the "Form W" process in which crosslinking occurs while the cotton is in an aqueous solution. Sometimes the surface areas retained appeared to be more a function of the method of processing than of the resultant add-on of the crosslinking agent. Analyses of complete isotherms obtained on a few of these samples showed a loss of area in the smallest pores which results in a less homogeneous pore-size distribution.

Also of interest are the findings: (1) That fabric which has been slack mercerized and air dried before reswelling in water for sample preparation had 70 percent more surface area than a fabric similarly treated except that it was mercerized and air dried under tension by holding to constant dimensions; (2) drying any fabric after treatment with mercerizing strength sodium hydroxide before reimmersion in water for sample preparation greatly reduced the surface area available to nitrogen; and (3) that the surface area of decrystallized cottons was not only sensitive to drying, but also to the temperature of the exchange liquids used during sample preparation.

SECOND SESSION: E. V. Painter, Johnson and Johnson, Chairman

PARAMAGNETIC RESONANCE SPECTRA OF COPPER COMPLEXES OF CELLULOSE

[Summary]

by
J. C. Arthur, Jr., P. J. Baugh, O. Hinojosa, and T. Mares
Cotton Chemical Reactions Laboratory
Southern Utilization Research and Development Division

(Presented by J. C. Arthur, Jr.)

Cupriammonia dihydroxide and cupriethylenediamine dihydroxide have been used as solvents for cotton cellulose in determining the intrinsic viscosity for the calculation of molecular weight. It has been suggested that cellulose complexes with these copper-containing compounds to yield colloidally dispersible products. Neither the nature of the chemical bonds involved in the proposed complexes nor the extent of definition of the proposed complexes have been experimentally shown. Copper-containing compounds are also used in improving the weather resistance of cotton. The question of whether chemical bonding occurred between these copper compounds and cellulose or if only deposition of these compounds within the cotton fibers was involved has not been resolved.

The paramagnetic resonance spectra of complexes of fibrous cotton cellulose and cupriammonia dihydroxide or cupriethylenediamine dihydroxide under various experimental conditions were determined. The spectra of both complexes with cotton cellulose were almost identical at -100° and 25° C. The spectrum of the complex of cupriethylenediamine dihydroxide with cellulose was stable to temperatures as high as 100°. The line widths of hyperfine components of the spectra for both complexes were about 180 gauss. Cotton fibers were combed and aligned with their axes (lengths) parallel to the pole faces of the magnet; after formation of either of the complexes with cellulose, a minimum of hyperfine structure of the spectra was observed. A maximum of hyperfine structure was observed when the complexed fibers were aligned with their axes perpendicular to the pole faces of the magnet. For a complex of cupriethylenediamine dihydroxide--cellulose the g-value (center of spectra) for the parallel alignment was 2.2127 and, for the perpendicular alignment, 2.0476. It was suggested that the alignment of most of the copper complex was with its axis of symmetry at a maximum angle to the axes of the cotton fiber, when the axes of the fibers were parallel to the pole faces of the magnet.

When ramie was used instead of cotton, cellulose, the hyperfine structure with fibers aligned parallel to the field showed no contribution of components centered at a parallel field. To obtain maximum swelling, the fibers were treated with 18 percent NaOH and then with cupriethylenediamine dihydroxide (0.001 M)--NaOH(7 percent). The sharpness of the intensity/rotation peak was greater for ramie than for cotton cellulose II. This showed an apparent increase in the alignment of the complex. When cotton cellulose I or II was used with no excess NaOH, then the differences in spectra due to the perpendicular and parallel orientations of the fibers in the magnetic field were not well-defined. The increased differences in spectra due to orientations of the fibers in the field were obtained on treatment of the fibers with NaOH. Mercerized ramie showed the greatest differences between the intensities of the components centered at a perpendicular field in the parallel and perpendicular positions of the fiber axes to the magnetic field. In the parallel alignment since no contribution from components centered at parallel field was shown, the angle of orientation of the axis of symmetry of the copper complex was very close to 90°. We see only the complex with axes of symmetry perpendicular to the fiber axes contributing to the spectrum. Rotation of the magnetic field until it was perpendicular to the fiber axes gave a spectrum with both sets of components. Rotation about the fiber axes in the latter position showed no change with intensity of the two sets of components. These changes in spectra with orientation of the complex fibers in the magnetic field gave a measure of the degree of orientation of the crystallites in the fibers of cotton cellulose I, II, and ramie.

The work was extended to show that little, if any, chemical bonding occurred between several copper-containing compounds (copper borate--zirconyl acetate, cupric chloride, copper sulfate, cupric hydroxide, cupric acetate, and copper-8-quinoliniolate), used in improving the weather resistance of cotton, and cellulose.

DISCUSSION

Question: Would you comment a little on the configuration of the cupriethylenediamine dihydroxide complex of cellulose and on the structure of the complex?

J. C. Arthur, Jr.: Cotton fibers were combed and aligned with their axes parallel to the magnetic field; after formation of either of the complexes with cellulose, a minimum of hyperfine structure of components centered at $H_{,,}$ was observed. A maximum of hyperfine structure of these compounds was observed when the complexed fibers were aligned with their axes perpendicular to the magnetic field ($B_{,}$). The opposite was true for components centered at $H_{,}$. For a complex of cupriethylene diamine dihydroxide--cellulose at 25° C. and at high pH $g_{,,}$ was 2.2127 and $g_{,}$, 2.0476. It was suggested that the alignment of most of the complex was with its axis of symmetry at a maximum angle, that is, perpendicular to the axes of the cotton fiber, when the axes of the fibers were in the parallel alignment.

LONG-LIVED FREE RADICALS IN GRAFT COPOLYMERIZATION REACTIONS OF COTTON

[Summary]

by
J. C. Arthur, Jr.
Cotton Chemical Reactions Laboratory
Southern Utilization Research and Development Division

On interaction of cobalt-60 gamma radiation with the cotton cellulose molecule, it can be concluded that long-lived free radical sites are formed within the molecular lattice. Further, some of the free radical sites are formed within regions of the cellulose molecule that are inaccessible to moisture and aqueous solutions of acrylonitrile and other reagents, even after long periods of time.

From the Electron Spin Resonance (ESR) spectra of irradiated cellulose I and II, containing regain moisture, or after being contacted with an aqueous solution, long-lived free radical sites were apparently formed at carbons C_5 and C_6 of the anhydroglucose unit. For cellulose I a fairly symmetrical three-line spectrum was obtained. The equivalent hydrogens on C_6 could split a radical spectrum into three lines, if the radical site was on C_5 or C_6. A free radical site could be formed on C_5 by dehydrogenation or on C_6 by dehydroxylation or by dehydrogenation of the OH group. Due to the delocalizing energy available from the oxygen attached to C_5, it is likely that the long-lived radical site in cellulose I containing moisture was at C_5.

For cellulose II a five-line spectrum was obtained. Considering the chemical composition of cellulose II, there is no obvious position for a free electron that could split with four protons to give five lines. It was concluded that the five-line spectrum resulted from the summation of two or more spectra, probably two three-line spectra having an overlapping principal line at about the same value in the magnetic field. For both cellulose I and cellulose II the unit cell is monoclinic and belongs to space group P2; however, there are some differences in cell dimensions, particularly a lower β angle in cellulose II than in cellulose I. Consequently, in cellulose II the anhydroglucose units are closer together and in a more parallel alignment than those in cellulose I. In cellulose II the hydrogen bonds originally present in cellulose I could be rearranged on mercerization to increase the delocalizing energy at C_6. This would increase the possibility of a long-lived free radical site being formed at C_6, by one of the processes indicated above, to give a three-line spectrum. The summation of the two three-line spectra from free radical sites at C_5 and C_6 would give a five-line spectrum.

When cellulose I and II with low-moisture contents were irradiated, similar ESR spectra were obtained. On addition of water to the irradiated celluloses, their ESR spectra changed to give the three-line spectrum for irradiated cellulose I and the five-line spectrum for irradiated cellulose II. Apparently in the irradiated celluloses with low moisture contents a long-lived free radical site was formed which gave a strong singlet spectrum added to the three-line or five-line spectra to give the observed spectra. A strong singlet spectrum could result on the formation of a free radical site on the oxygen in the cellulose chain attached either to C_1 or C_4.

During the graft copolymerization reaction, the ESR spectrum of the irradiated cellulose I changed from a three-line spectrum to apparently a single-line spectrum. The single-line spectrum could indicate that the initial free radical site on C_5 had reacted with water and/or acrylonitrile and that the radical being observed after 17 hr. was on the end of the polyacrylonitrile chain. The extent of graft copolymerization, as measured by the percentage of graft copolymer formed, was dependent on the radiation dosage, that is, free radical concentration.

Experimental data would support the free radical mechanisms generally proposed for the post-irradiation reaction of cellulose with vinyl monomers. The initiation of the reactions would depend on the accessibility of the long-lived free radical sites in the irradiated cellulose. In aqueous systems, as well as probably for other solvent systems, there would be competition between solvent and monomer for the free radical sites. It has been reported that in post-irradiation reactions the degree of crystallinity of the cellulose lattice is inversely related to the rate of termination of the graft copolymerization reaction with vinyl monomers.

When the objective is to produce a graft copolymer of cotton cellulose, particularly with high molecular weight copolymer deposited within the fibrous structure, the use of long-lived free radicals makes possible a two-step process. In the first step the cotton, containing regain moisture, would be irradiated in air to form the free radicals. In the second step the activated cotton would be reacted in a wet process in a solution containing the vinyl monomer. Possible applications to large-scale processes are discussed.

DISCUSSION

Question: Couldn't functional groups be introduced into cellulose at specific desirable sites and chosen so they will result on irradiation in radicals with half-lives long enough to be used to initiate graft polymerization? This method of graft polymerization could result in large changes in properties at low graft polymer contents.

J. C. Arthur, Jr.: Natural cellulose contains functional groups, namely, hydrogen, which on irradiation give radicals with long half lives. By changing the lattice type and considering energy relationship in the anhydroglucose unit, these radicals can be located at C_5 or C_6. Radicals formed at other locations would lead primarily to degradation of the cellulose molecule. Localization of high energy in the cellulose molecule is chemically nonspecific. The latter part of the question expresses a speculative hope which has only been partly realized with any graft polymer of cellulose.

Question: Would the techniques used by Douglas J. Bridgeford, Research and Development Department, Tee-Pac, Inc., Danville, Ill., be a method of grafting at specific desirable sites in the cellulose molecule?

J. C. Arthur, Jr.: The basic chemical principles of ceric ion initiated oxidation or formation of transient radicals on C_2 or C_3 of cellulose molecule or oxidation of dihydroxyl containing alcohols are a matter of technical record in several journals and books. The use of Fenton's reagent (ferrous ion/hydrogen peroxide) to generate hydroxyl radicals which, of course, could abstract accessible H-atoms from the cellulose molecule to generate a radical site has been reported in the basic chemical literature. No experimental evidence has been published showing the existence of these hydroxyl radicals directly.

QUICK-DRYING COTTON

[Summary]

by
N. R. S. Hollies
Harris Research Laboratories
Washington, D. C.

Measurement of Drying Properties

The drying properties of a variety of cotton fabrics have been measured directly by weighing samples periodically from the dripping wet state to equilibrium in a conditioned atmosphere. In agreement with the work of Fourt[1], Coplan[2], and Steele[3] it has been established that over a major portion of the drying time, water evaporates freely from the fabric surface at a rate depending primarily on the fabric surface area rather than on its structure. Thus, the total drying time is determined mainly by the amount of water picked up by the fabric when wet, and this, indeed, varies greatly with the structure. In the current work the water content of a fabric after wetting and spinning in a washing machine has been found to give reliable and useful indication of the time it will take the fabric to dry.

Approaches to Quick Drying Systems

The drying times and water contents of cotton fabrics in the weight range of 3-1/4 (print cloth) to 10-1/4 (upholstery) oz./yd.^{2} have been measured with and without a variety of quick-drying finishes. Various combinations of crosslinking resins and water-repellent finishes have been found to reduce the water content and drying time of every cotton structure. For example, with a triazone resin and fluorocarbon polymer the water content (after spinning) of a 7-3/4 oz. duck has been reduced from 81 to 23 percent. Drying time of a jersey knit has been reduced from 5-1/2 to 3 hours by using a melamine-DHDMEU finish topped with a fluorocarbon-type water repellent.

1/ Coplan, M. J.
1953. Some Moisture Relations of Wool and Several Synthetic Fibers and Blends. Textile Res. J. 23: 897–916.

2/ Fourt, L., Snooke, A.M., Frishman, D., and Harris, M.
1951. The Rate of Drying of Fabrics. Textile Res. J. 21: 26–33.

3/ Steele, R.
1958. Factors Affecting The Drying of Apparel Fabrics. Part I: Drying Behavior. Textile Res. J. 28: 136–144.

Mechanism of Drying Action

Microscopic examination of the structures has shown that the finishing treatments reduce water-holding capacity of the fabrics by repelling water from the capillary shaped interior portions of the fabrics and yarns and by keeping water at the surface from which evaporation can readily take place. Some combination finishing treatments have been found to stabilize the fabric structure and wetting properties against repeated laundering. Hence, the resulting fabrics are as quick-drying after 15 laundering and tumble drying cycles as when initially finished.

A standard 5-3/4 oz. duck fabric was given a variety of combination finishes. Some of these, producing only moderate reduction in water content of the fabric, resulted in exceptional quick-drying properties. A close examination of the drying curves of these fabrics indicated that the drying process had been substantially modified. These treated fabrics of modified drying behavior suggest a number of new approaches for achieving quick-drying properties on a variety of cotton structures.

DISCUSSION

Question: During the drying of a fabric, is the mechanism by which moisture reaches the surface from the interior one of diffusion, wicking, or channeling? The nature of the finishes just described seems to rule out wicking or capillary action.

N. R. S. Hollies: Moisture transmission appears to be mainly by diffusion.

Question: One of the anomalies of your data may arise from not separating the water of imbibition and the capillary water. This is shown by the different behavior of different weaves at the same centrifuge rate. Drying requires drainage and you used a centrifuge which supplies energy. In natural drying, you have only the force of gravity to aid drainage. Drip drying is vastly different from centrifuging. Much water is held between yarns and fibers. Do you feel the fluorocarbon finish would assist drainage where the water is held in the fabric by a tight weave as in terry cloth?

N. R. S. Hollies: Tumble drying is more generally used than drip drying and corresponds to our method of measuring water content after centrifugating. The correlations we observed indicate the water of imbibition is not of major importance to the drying rate.

Question: Previous work has shown that the fiber diameter has an effect on water content and drying rate. Was this factor studied?

N. R. S. Hollies: No. It probably should be studied.

Question: How do the treated fabrics rate in regard to comfort?

N. R. S. Hollies: They have excellent feel and comfort and in many instances cannot be distinguished from the untreated fabrics.

Question: Did you observe that the drying rate became slower, as the last traces of moisture were removed, in the treated samples than in the untreated ones?

N. R. S. Hollies: This effect was pronounced in some of the treatments, but not in the optimum ones described.

Question: Did the treatments produce any change in hand or color?

N. R. S. Hollies: The levels of fluorocarbon applied were so low that only nominal changes occurred.

OIL-REPELLENT FINISH FOR COTTON BASED ON FLUORINATED AMINES

[Summary]

by
S. E. Ellzey, Jr., W. J. Connick, Jr., G. L. Drake, Jr., and W. A. Reeves
Cotton Finishes Laboratory
Southern Utilization Research and Development Division

(Presented by S. E. Ellzey, Jr.)

With the objective of rendering cotton fabric durably oil- and water-repellent, a program was initiated in which various types of polyfluorinated compounds were reacted with conventional multifunctional finishing agents and the resulting products were evaluated on cotton fabric. One of the systems chosen for exhaustive study was that based on reactions involving tetrakis(hydroxymethyl) phosphonium chloride (THPC) and fluorinated amines.

Although the literature contains reports of reactions between amines and THPC, characterization of the products has been of a limited degree and reactions with fluorinated amines apparently have not been reported.

A very brief study of the reaction of 1,1-dihydroperfluorooctylamine and THPC was carried out in which the ratio of amine to THPC was varied. Product composition apparently varied little with reactant ratio.

The product from the 1:1 reaction, when applied to printcloth from organic solvents, gave high oil repellency ratings but low water repellency ratings. The useful lifetime of a solution of this product in most organic solvents is short, since precipitation of a gummy product begins about 2 to 3 hours after preparation. Emulsions of excellent stability can be prepared using up to 10 percent solids.

Application of either solutions or emulsions containing the THPC-amine product causes high strength losses after the usual drying step and heat cure. Inclusion of triethanolamine in the treating bath hastens the precipitation of gummy product.

An investigation of various methods of curing with ammonia was carried out. The best balance among add-on, oil repellency, tearing strength, and durability of repellency was achieved by treating the wet, padded fabric with ammonia gas for 3 min., followed by drying at 80° C.

Samples of printcloth were treated in small pilot-plant runs with 2.5 percent and 5 percent emulsions and textile properties were evaluated. Abrasion resistance of fabrics treated with the 5 percent emulsion (2.3 percent add-on) is about 30 percent lower than the control, while samples treated with the 2.5 percent emulsion (0.9 percent add-on) have about the same value as the control. Although tearing strength is slightly lower, breaking strength is slightly greater than the control. Elongation and moisture content and regain are slightly increased but air permeability is reduced about 25 percent by the treatment. Wrinkle recovery is little affected. Durability of the repellency to laundering is good but extraction with perchloroethylene lowers oil repellency significantly.

Synthetic aqueous and oily soils were used to test the soiling properties of the THPC-amine finish and to compare it with three commercially used fluorocarbon finishes. Reflectance values before and after soiling and after one home laundering were used as measures of the degree of soil pickup and release. Reflectance values before and after laundering in the presence of a large amount of added soil were used to judge the soil redeposition properties of the fabrics. The degree of aqueous soiling of the THPC-amine finish was comparable to that of one commercial finish but higher than the other two. Aqueous soil release followed the same general pattern. The performance of the THPC-amine finish was better than the commercial finishes in oily soiling tests. All finishes were comparable in the aqueous redeposition tests but the commercial finishes were poorer in the oily soiling redeposition tests.

DISCUSSION

Question: What is the yield of $C_7F_{15}CH_2$-NH_2 from the $NaBH_4$ reduction of the corresponding amide?

S. E. Ellzey, Jr.: The yield was low--about 40 percent--and the fluorinated alcohol was the major product.

Question: Is the finish fire-retardant?

S. E. Ellzey, Jr.: No.

Question: Is the NH_3 cure necessary?

S. E. Ellzey, Jr.: It gives better durability and lessens the strength low in the treated fabric.

Question: What is the fate of the chloride ion?

S. E. Ellzey, Jr.: It has formed the corresponding fluorinated amine hydrochloride, although this is not certain.

Question: Has THPOH been tried instead of THPC?

S. E. Ellzey, Jr.: No.

Question: What is the effect of sunlight on the finish?

S. E. Ellzey, Jr.: After 1 month of exposure, there seemed to be no noticeable change in fabric color.

Question: What is the pH of the emulsion?

S. E. Ellzey, Jr.: About pH_2.

Question: Has it ever been neutralized before use?

S. E. Ellzey, Jr.: No.

Question: Is the water repellency of the treated fabrics increased by thoroughly washing and drying?

S. E. Ellzey, Jr.: No.

Question: Is the object to obtain a hydrophilic finish?

S. E. Ellzey, Jr.: The presence in the finish of hydrophilic groups probably accounted for the fact that the treated fabric had some--but very slight--water repellency, although the treated fabric was more water-repellent than untreated cotton.

A DURABLE FLAME RETARDANT FINISH FOR COTTON

[Summary]

by

J. V. Beninate, E. K. Boylston, G. L. Drake, Jr., and W. A. Reeves
Cotton Finishes Laboratory
Southern Utilization Research and Development Division

(Presented by J. V. Beninate)

The Southern Utilization Research and Development Division has been actively engaged in research to develop durable flame-retardant finishes for cotton for about 15 years. Several durable finishes have resulted from this effort, and those based on tetrakis(hydroxymethyl)phosphonium chloride, (THPC), first announced by Reeves and Guthrie in 1953, perhaps have had greatest commercial acceptance.

Although these flame retardants are being used commercially, there is a need to develop better systems which are applicable to lightweight fabrics, and which do not lower the tearing strength qualities or change the hand of the fabrics.

This paper presents a new flame-retardant finish for cotton which is based on the reaction product of THPC and sodium hydroxide. This product was prepared by the method of Filipescu in which a methanolic sodium hydroxide solution is added to solid THPC with mixing at room temperature or below. The rate of addition of sodium hydroxide was controlled to maintain a pH of 7, or lower, in the solution. The product remaining in the methanol after filtration is believed to be an equilibrium mixture of tetrakis(hydroxymetyyl)phosphonium hydroxide (THPOH) and tris(hydroxymethyl)phosphine (THP).

A pad, dry, ammonia cure technique is used to apply the finish to cotton. A typical treatment consists of padding fabric through a 20-percent solution of THPOH, drying the fabric to approximately 10-percent moisture, using heated tenter frame or roller oven, and then exposuring to ammonia vapors. The ammonia vapors react immediately with the THPOH forming highly insoluble flame-resistant polymers inside the cotton fabric.

Cotton fabrics of from 2 oz./sq. yd. to 8.5 oz./sq. yd. have been rendered flame resistant by this treatment with little or no change in the strength properties or hand of the fabrics. Studies were made on the effect exposure time in the ammonia gas of impregnated fabrics had on the efficiency of the process. Generally, longer exposure time results in greater efficiency in the treatment. Resin add-ons generally increased as exposure time was increased. Increasing the resin add-on on the fabric increased the degree of flame resistance and the strength properties of the fabric were essentially unaffected.

The durability of the finish to laundering was studied. Printcloth samples with resin add-ons of about 16 percent and 8.5 oz. sateen samples with add-ons of about 12 percent remained flame resistant after 30 home laundering cycles.

Fabrics were treated with a THPOH solution which had aged for 6-1/2 weeks and the efficiency of the treatment and flame resistance were essentially the same as treatments using freshly prepared THPOH.

Drying of fabrics wetted with THPOH appears to be critical especially when the very lightweight fabrics are used. Overdrying of the wetted fabric before exposure to ammonia vapors can lead to greatly reduced efficiency. Overdrying of the fabrics apparently causes a conversion of the THPOH to a less reactive compound. Optimum drying times of from about 15 seconds to several minutes were established for fabrics of varied weights and it was found that a moisture content of about 10 percent was optimum to give greatest efficiency.

Short yardages of various weight fabrics were satisfactorily processed on a semi-pilot-plant scale, and the properties of the treated fabrics were evaluated.

This THPOH finish has several advantages over previous flame retardants in that it is applicable to lightweight fabric and does not cause any disagreeable change in hand or strength. This finish does not impart any crease resistance to cotton and the yellowing associated with other flame-retardant finishes when applied to cotton and bleached is also associated with this finish, but to a lesser degree.

DISCUSSION

Question: Has this finish been applied to napped materials?

J. V. Beninate: Yes, this finish was successfully applied to light blanket and flannel fabrics.

Question: How does THPOH compare with APO or conventional flame retardants with respect to cost?

J. V. Beninate: Since THPOH is a derivative of THPC it should be in the same price range. Assuming that THPC is being used in conventional flame retardants, then the THPOH finish would be in the same range costwise.

Question: What is the effect of this finish on wet wrinkle recovery? What effect on cuene solubility?

J. V. Beninate: We have noticed no increase in wet wrinkle recovery angles of fabrics treated with THPOH; however, a slight increase in conditioned wrinkle recovery was obtained in some cases. We have not as yet studied the solubility of the treated fabrics in cupraethylenediamine solution.

Question: Of major concern to industry is the application of flame retardants to brushed or napped materials. Are you in tune to what industry wants?

J. V. Beninate: We have applied this finish as well as other durable flame retardant finishes to a variety of napped materials. We are also studying the application of less expensive semi-durable finishes to these materials. We have been in contact with manufacturers who produce blankets, sweat shirts, flannel fabrics, etc., and are aware of their needs.

THIRD SESSION: F. Fortess, Celanese Fibers Marketing Co., Chairman

HIGHLIGHTS OF NEW CHEMICAL TREATMENTS FOR WOOL

[Summary]

by
W. L. Wasley
Western Utilization Research and Development Division

Studies on the chemical finishing of wool at the Western Utilization Research and Development Division, Albany, Calif., at present include treatments for stabilization against felting shrinkage, treatments to improve flat setting and creasing, and treatments to provide increased resistance to water and oil.

Although so much has been said about wool shrinkproofing over the last several years, it is still receiving more effort than any other single subject in our Wool Research Laboratory and in many other textile laboratories around the world. The International Wool Secretariat is giving added impetus to research and development work on shrinkproofing with their insistance that wool must be machine washable if it is to carry their "Wool Mark" after 1968. As for our WURLAN (interfacial polymerization) treatment, it was compared recently with six other shrinkproofing treatments in an international round-robin test supervised by the German Textile Research Laboratory in Aachen. In this test, our WURLAN treatment proved to give the best shrinkproofing under rigorous washing conditions.

Recent improvements in WURLAN processing have included the use of an improved solvent system and a lower cost diamine. It has also resulted in improved hand and improved stability in all-wool knit goods.

The outstanding progress being made in no-iron cottons gives added importance to the search for no-iron finishes for wool.

By far the best machine washable, tumble dry, no-iron performance that we have seen in any garments containing wool has been achieved with fabrics made of wool and cotton. We have applied the WURLAN treatment to 50/50 wool/cotton fabrics to stabilize them against felting shrinkage. After the WURLAN treatment, a delayed-cure cotton-type permanent-press finish was applied, that is, dihydroxy dimethylol ethyleneurea (DHDMEU). When this is cured, the cotton component contributes its excellent no-iron effect to the fabric. Experimental shirts and slacks made of cotton/wool fabrics and treated in this manner at our laboratory have gone through as many as 40 washes and tumble dryings without the need for any ironing whatever.

It was found that DHDMEU, under the conditions used, did not make the wool shrink-resistant and there was no significant crosslinking of the wool. The DHDMEU and the heat of curing caused the wool to yellow appreciably.

Recent exploratory work in our group has shown that visible or UV radiation can initiate polymerization in wool in the presence of suitable photosensitizers. High energy radiation also initiates graft polymerization in wool, as shown by work under one of our research contracts.

Progress is also being made in new fluorochemical finishes which provide fabrics with resistance to oil and water. Poly(heptafluoro isopropyl vinyl ether), with a molecular weight of 70,000, has a critical surface energy lower than that of Teflon. It is completely stable toward acids and alkalies and should give durable oil and water repellency in many applications.

DISCUSSION

Question: Was the vinyl ether polymer stable to washing?

W. L. Wasley: Yes.

Question: What were the oil (or water) repellent ratings?

W. L. Wasley: The ratings were for moderate repellent action--about a value of 5.

Question: Is it easier to treat piece goods or flat goods using the WURLAN treatment? Is HCl liberation a problem?

W. L. Wasley: There is no significant difference in treatment by piece goods or flat goods. Liberated HCl is not a problem, it is neutralized by the added base.

Question: Has SURLAN T been tried and how does it compare with the WURLAN treatment?

W. L. Wasley: We have looked at it several years ago. At least 2 years ago WURLAN was a better treatment. Possibly further work by DuPont has produced an improvement.

Question: Have you used non-yellowing crosslinking agents, such as the carbamates, on wool?

W. L. Wasley: No. We have used DMEU.

Question: Concerning fabrics of one-third wool, one-third polyester, and one-third cotton blends, is it true that there is no need to shrinkproof the wool? Please comment on shrinkproofing of blends.

W. L. Wasley: You are probably correct, but we are working with blends containing a higher percentage (about 75 percent) of wool. Blends with percentages of wool over 50 percent will generally require shrinkproofing of some type.

Question: Plants don't like fabrics with three components in the blend. Do we know any more of the mechanism of the WURLAN treatment? Is it surface or does it affect internal structure of wool fiber to change moisture absorption for example?

W. L. Wasley: Polymer is on surface and water absorption of treated and untreated is essentially the same. Surface friction increases in both directions, appears to be a surface affect.

Question: An ill effect of the resin on the wool was noted. Have you tried pretreatment of cotton and then mixing?

W. L. Wasley: No, that's a good idea.

SOME OBSERVATION ON THE ELASTIC BEHAVIOR OF RESIN-TREATED COTTON BATTING AT TEMPERATURES BETWEEN 135° AND 170° F.[1]

by
N. B. Knoepfler and W. T. Gentry, Jr.
Southern Utilization Research and Development Division

(Presented by N. B. Knoepfler)

Introduction

Many researchers have carried out studies on the effects that can be achieved by the treatment of cotton with a wide variety of resins to attain wash-wear or durable-press characteristics. Each of these phenomena are manifestations of elasticity and the ability to recover from deformation.

The research reported herein was aimed at the development of an improved cotton batting product which would exhibit enhanced elasticity and dimensional stability. To confer such product performance characteristics on the cotton fibers and on the fiber array, both thermoplastic and thermosetting resins were found to be needed.

The outgrowth of the research was the development of a process for the spray treatment of unscoured, unbleached cotton fibers while they are openly arrayed in a web form. The treating formulations used generally consist of 20 percent solids, usually equally divided between thermoplastic and thermosetting resin components, with the optimum wet add-on being 100 percent based upon the weight of the cotton fibers. The treated fibers are then lapped conventionally, dried, and cured (8, 9, 10, 11, 12, 13, 14, 15).

The products demonstrate considerably enhanced ability to recover (90+ percent in 4 min.) from cyclic compressive deformation when compared to untreated cotton batting (70+ percent in 4 min.). The tensile strengths of the products are from 5 to 15 times that of untreated cotton batting.

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[1] A cooperative research undertaking of the Textile Waste Association, The National Cotton Batting Institute, The National Cottonseed Products Association, The Foundation for Cotton Research and Education, and the U. S. Department of Agriculture.

Some unusual behavioral characteristics were, however, observed in the products when they were evaluated for their ability to recover from sustained compressive loading at elevated temperatures. This, too, is a manifestation of elasticity.

The ability of a cushioning material to recover from sustained compressive stress is usually measured by a modification[2] of the ASTM test method D 1564-62T (method B) (1). The procedure used requires that the sample be compressed between parallel plates, figure 1, to one-half its original thickness. The compressed sample is then held at 158° F. for 22 hr., after which it is removed from the jig and allowed to recover at ambient laboratory conditions of 72° 65-percent relative humidity. Thickness measurements are made at selected intervals to trace the recovery pattern, with the 30-min. reading generally reported as percent set calculated from the following equation:

$$S = \frac{T_0 - T_1}{T_{1/2}} \times 100$$

Where T_0 = original thickness, T_1 = measured thickness, $T_{1/2}$ = compressed thickness, S = percent loss in thickness. For the purpose of this report, the term "heat-set" will be used to designate the loss in thickness resulting from the above calculation.

An analysis of the data on over 700 samples of both treated and untreated conventional cotton batting (2 lb./cu. ft. density), using the modified ASTM procedure[3], demonstrates that it is practically impossible to meet a heat-set value of 30 percent established by an automobile manufacturer[4].

Treated cotton batting samples generally have 158° F. heat-set values between 50 and 60 percent. Occasional samples have heat-set values in the low 40 percents, while others have heat-set values in excess of 70 percent. In comparison, untreated cotton batting samples have heat-set values of 35 percent. The consistently higher heat-set values for the resin-treated samples prompted this investigation into the causes for the observed phenomenon. Four temperatures--135°, 145°, 158°, and 170° F.--were selected to determine how the temperature of the storage period affected the heat-set. In addition, the moisture contents of the samples after 22 hours' exposure to the test temperatures listed above were obtained, and the

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[2] Chrysler Corporation Laboratory Procedure 463KB-5-01.

[3] See footnote 2.

[4] Chrysler Corporation Material Standard MS-JZ-5-23 change C.

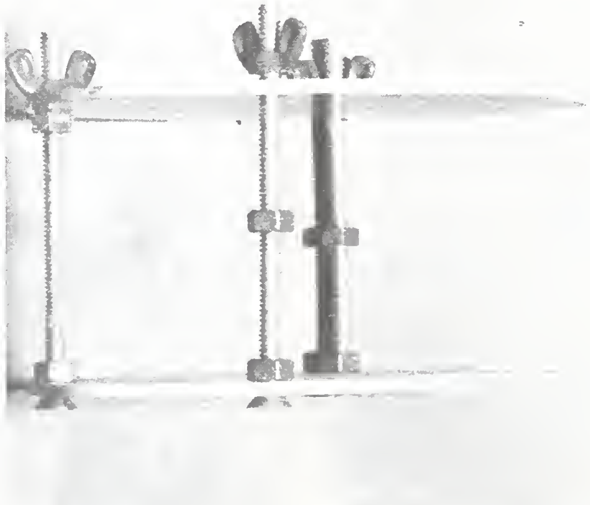

Figure 1.--Jig for heat set test.

effect of relative humidity during recovery was studied.

Sample Preparation

The samples for this work were produced on a Davis and Furber sample card equipped with garnett wire. The feed material consisted of a preblend of fibers, 60 percent first cut linters and 40 percent textile wastes, in the form of a 16 oz. /yd. picker lap. The lap was split in half lengthwise and each half then doubled to assure maximum uniformity in feed. The feed rate was maintained at 80 g. /min.

As the web was discharged from the card (garnett), it was sprayed with the treating formulation to a wet add-on of 100 percent by weight of the cotton fibers. The treating formulation contained 20 percent solids made up of equal amounts of thermoplastic and thermosetting resins. In cases where the components were being evaluated independently they were applied from formulations containing 10 percent solids.

The thermosetting resins used in the formulation were methylated methylol melamines, urea formaldehydes, urons, dimethylol imidazolidones, and phenol formaldehydes. The thermoplastic resins used were vinyl acetates, vinyl acrylates, vinyl acetate-acrylates, vinyl chlorides, styrene butadienes, styrene acrylates, and a neoprene. Combinations of each thermosetting with each thermoplastic resin were evaluated in almost every case. The treating formulations included dicyandiamide as the buffer and magnesium chloride as the catalyst, except where dimethylol imidazolidones and phenol formaldehydes were used.

For the former, the manufacturer's proprietary zinc chloride catalyst was used, while for the latter no catalyst was required. All treating formulations contained approximately 1 percent by weight of reactive cotton dyes for the visual assessment of the uniformity of application.

The spray damp web was collected on a rotating reel until 20 webs were built up. The wetted, in-process, material was removed from the reel, then dried and cured in a continuous pilot-plant oven (5). Drying temperature was 200° F. over a 4- to 6-minute time interval. Curing temperature was 300° to 320° over a 2- to 3-minute time interval. During drying and curing, air pressure differential across the sample was maintained at 0.3 inches of water.

Control samples of untreated cotton batting were produced as outlined above with water being substituted for the treating formulation, following which the samples were dried at 200° F. in 4 to 6 minutes and then subjected to 300° to 320° curing conditions for 2 to 3 min.

The experimental samples of batting were usually between 7/8 and 1-1/8 in. thick and had densities of 1.8 to 2.2 lb. /cu. ft.

After curing, the samples were equilibrated at ambient pilot-plant conditions for at least 48 hr. before evaluation work was initiated. At the conclusion of this storage period test samples 4-1/2 in. in diameter (15.97 sq. in. area) were die cut from the production samples. The samples were then equilibrated at 70° ± 2° F. and 65° ± 2 percent relative humidity for 24 hr. before the tests for performance under compressive stress at elevated temperature were run.

Results and Discussion

The heat-set values obtained by testing samples of cotton batting treated with formulations containing equal parts of thermoplastic and thermosetting resins where methylated methylol melamine is the thermosetting component are shown in figure 2. For purpose of comparison, the data obtained at each of the four test temperatures were connected by straight lines. This should not be interpreted as meaning that a sharp peak would have been obtained if other values had been obtained in close proximity to the selected test temperatures. In all probability the lines between points would assume a curved configuration with a gentle approach to the observed maximum.

An analysis of this data shows that while there are some differences in the magnitude of heat-set for the various thermoplastic resins in combination with methylated methylol melamine, a maximum in the heat-set value is always found at the 158° F. test temperature. This unusual symmetry of the heat-set performance suggests that there is a second-order process point (2) between 145° and 170° for cotton fibers that have been treated with thermoplastic and thermosetting resins. A maximum in the heat-set value at the test temperature of 158° is also evident for samples that have been treated with equal parts of thermoplastic resin and either methylol imidaxolidone or modified urea formaldehyde resin (figure 3). Because of the varied nature of the chemical struc-

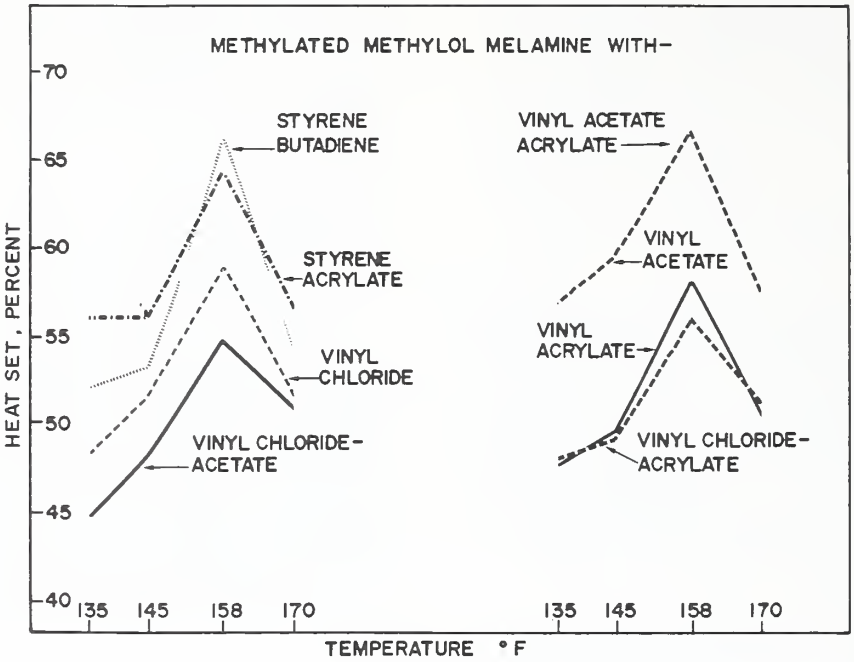

Figure 2.—Percent heat set vs. temperature of test.

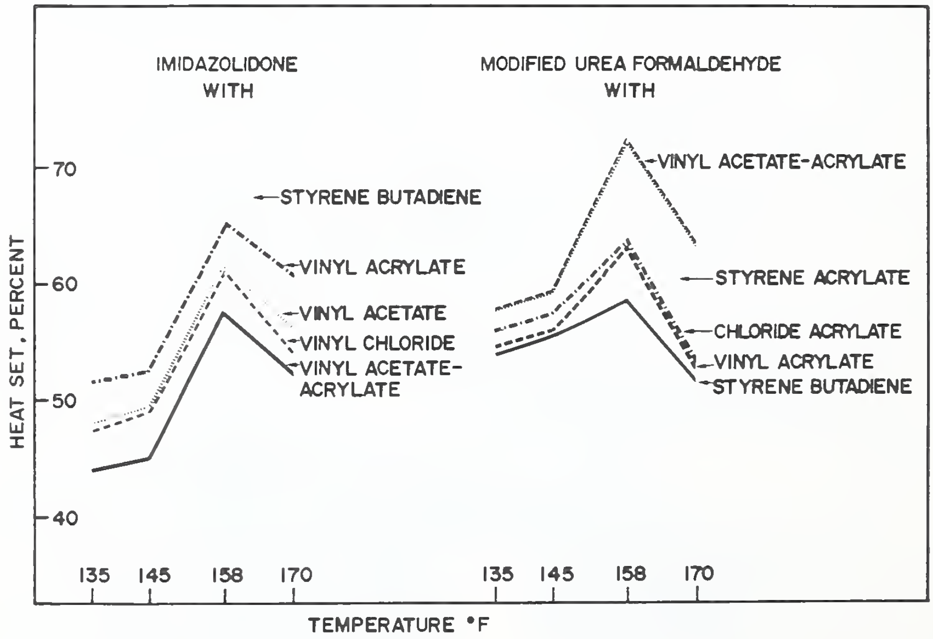

Figure 3.—Percent heat set vs. temperature of test.

tures of both the thermoplastic and thermosetting resin components, this conformance to a maximum in the heat-set performance at 158° cannot be explained on the basis of structure. There are indications in the literature that combinations of thermoplastic and thermosetting resins in the presence of a cotton substrate do not yield products that exhibit an average performance between that which would be achieved by any of the components alone (6, 16). Systems containing multiple polymers usually demonstrate a depressed transition point below that of the component with the lowest transition point (6).

The literature discloses that a number of the thermoplastic resins of the type used in this investigation have critical or transition points within 40° F. of the 158° test temperature (2, 20), and that a variation of this magnitude in second-order processes or glass transition points was not unusual (7). While there do not seem to be any transition points reported for cotton near the 158° temperature, some unusual behavior has been reported at 160° for chemically modified cotton (4).

Figure 4 shows the curve obtained when heat-set data were plotted for a sample of untreated cotton batting that previously had been sprayed to a 100 percent wet add-on with water, then dried and subjected to curing temperatures of 300° to 320° F. 2 to 3 min.

The shape of this curve is somewhat different to that obtained with the resin-treated samples in that a broad maximum occurs between 145° and 158° F. This indicates that some type of second-order process might be taking place in the cotton itself.

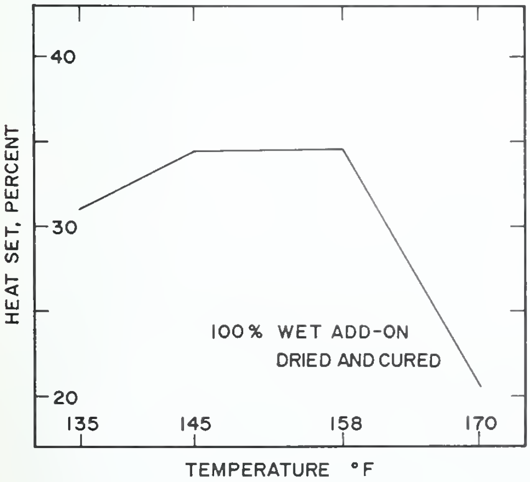

Figure 4.—Heat set performance of cotton sprayed with water.

In order to separate the effects of the thermoplastic resin from that of the thermosetting resin in the presence of cotton, a series of samples made using only one or the other of these components at the 10 percent add-on level were tested for heat-set at the four selected temperatures. The data obtained are shown in figures 5 and 6. It will be seen from a comparison of these figures that two distinct types of curves resulted from a plot of the heat-set against temperatures. Figure 5 shows that most of the thermoplastic resins tested exhibited a peak at the 158° F. temperature following much the same pattern as was observed in figures 2 and 3 for combinations of thermoplastic and thermosetting resins. In contrast, the thermosetting resins shown plotted in figure 6 tend to gradually increase in heat-set up to 158° F. at which point there is a sharp upward change in slope. Each of these data may be interpreted as second-order process phenomenon.

It should be noted that both polyurethane and vinyl acetate-acrylate exhibit heat-set characteristics more like the thermosetting than thermoplastic resins. If these data from figure 5 are, in turn, compared with the data shown in figures 2 and 3, it can be seen that vinyl acetate-acrylate gave high

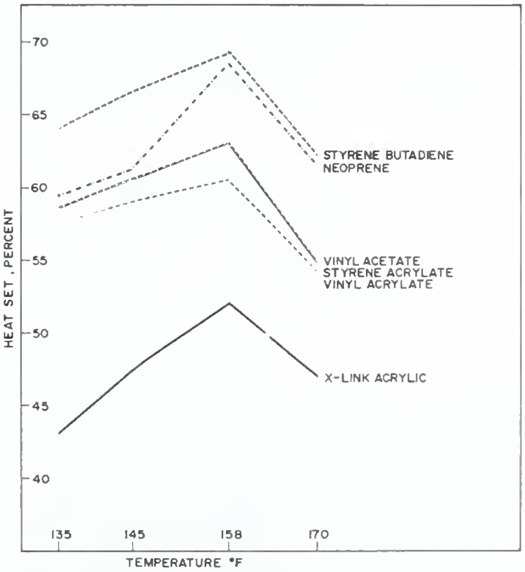

Figure 5.—Heat set vs. temperature for thermoplastic resins. 10 percent solids in spray, 100 percent wet add-on, dried and cured.

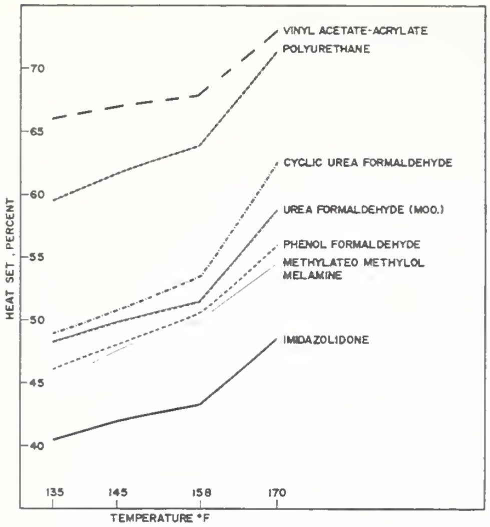

Figure 6.--Heat set vs. temperature for thermoset resins. 10 percent solids in spray, 100 percent wet add-on, dried and cured.

heat-set values when used in combination with either methylated methylol melamine or modified urea formaldehyde resins, and a low value when combined with dimethylol dihydroxy ethyleneurea. Conversely, styrene butadiene in combination with either methylated methylol melamine or dimethylol dihydroxy ethyleneurea gave high heat-set values, and low heat-set values when used with modified urea formaldehyde resin. These phenomenon are probably evidence of interactions between the components and the substrate or both--or possibly the presence of some type of mutual plasticization. Failure of the samples to recover more completely from compressional deformation at 158° F. may be attributed to plastic or elastic deformation of the resins used (18).

Plasticization as a factor in the behavior of the products was evaluated by adding external plasticizers to the formulation. Dioctyl phthalate, alkyl aryl phosphate, and a proprietary epoxy plasticizer were used. The plasticizers were added to the solution-suspension of the thermoplastic and thermosetting resins in the form of an emulsion. The emulsion was obtained by placing the calculated amount of plasticizer in a Waring Blendor with 50 to 100 cc. of water and 0.1 g. of trimethyl nonal then blending for 1 to 3 min. The amount of plasticizer added was based upon the weight of thermoplastic resin solids. Internal thermoplastic resin plasticization was also evaluated using samples of styrene butadiene, vinyl chloride, and vinyl acrylates selected based upon the manufacturer's property data sheets.

Figure 7 compares the performance of samples externally plasticized with alkyl aryl phosphate and dioctyl phthalate with a control containing the same formulation. The top two examples are the curves obtained from samples treated with methylated methylol melamine and vinyl acetate plus plasticizer. In each of these instances the addition of plasticizer, at the 5.5 percent and 7.3 percent levels with respect to thermoplastic resin solids in the formulation, increased the heat-set of the products.

Where methylated methylol melamine and vinyl acrylate plus plasticizer were used in the formulation, the addition of alkyl aryl phosphate as the plasticizer at the 5 percent level reduced the heat-set below the control at all test temperatures except 158° F., as can be seen from the bottom left family of curves. At the 6.7-percent level, alkyl aryl phosphate did not significantly affect the heat-set at 135° or 170° compared to the control, but did greatly increase the heat-set at 145° and 158°. From the lower right family of curves where dioctyl phthalate was used in a formulation containing methylated methylol melamine and vinyl acrylate, it can be seen that at the 5.0 percent level the heat-set was slightly lower than its control at 135° and 170° essentially equal to its control at 145°, but much higher than its control at 158°. At the 6.7-percent dioctyl phthalate level the heat-set values of all samples were higher than the control at all temperatures.

In figure 8 the data on heat-set are plotted against temperature for samples that contained different levels of epoxy plasticizer with the control containing the same formulation without plasticizer shown at the upper right. It can be seen from the curves at the upper left that at both the 10-percent and the 20-percent levels the addition of epoxy plasticizer lowered the heat-set values at all test temperatures. These samples, however, still demonstrated a maximum at 158° F. When the plasticizer was increased to 30 percent by weight of the thermoplastic resin solids, the peak was shifted to 145°, as shown in the lower left curve. At this level the heat-set values were higher than that of the control for all temperatures. At the lower right of figure 8 are the heat-set vs. temperature curves for samples containing

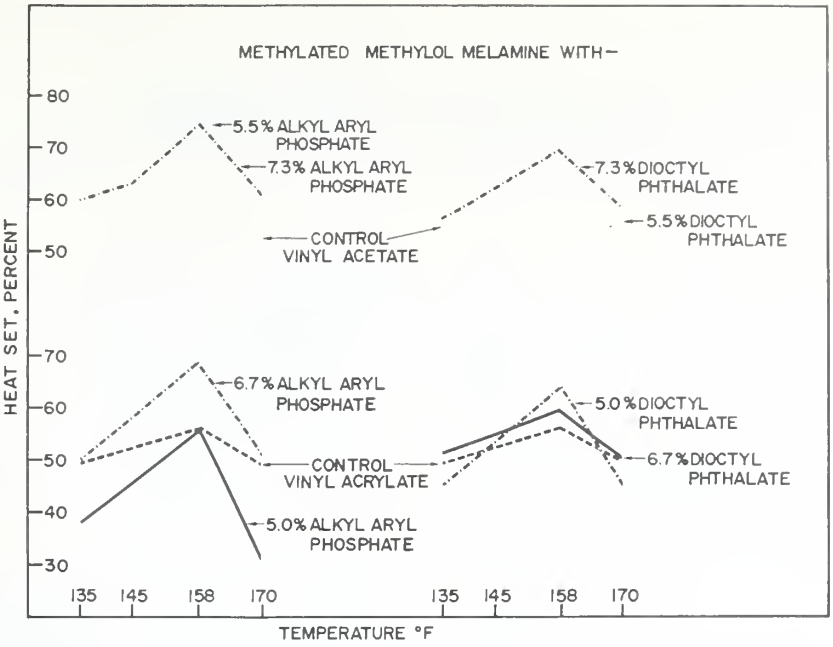

Figure 7.—Affect of added external plasticizer on heat set.

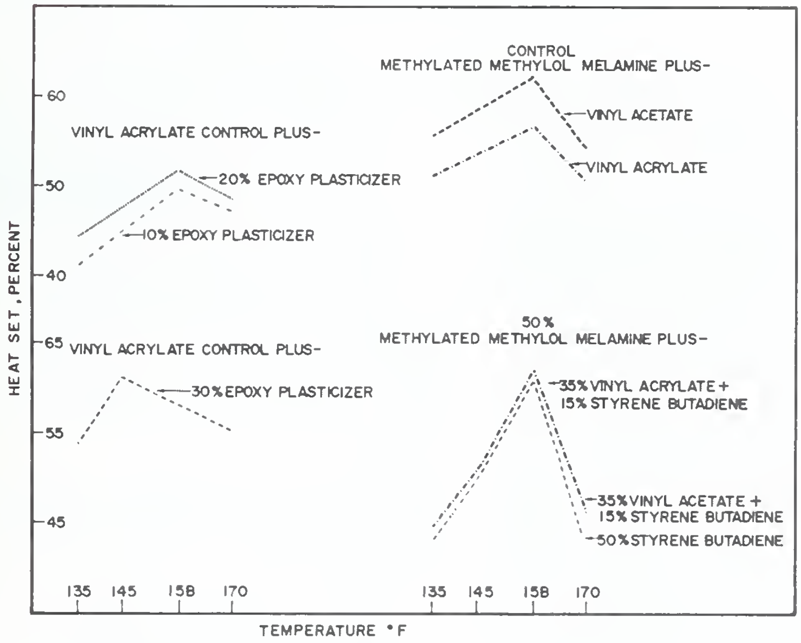

Figure 8.—Affect of plasticization of thermoplast on heat set.

styrene butadiene resins that were internally plasticized. Here again the maximum heat-set value is shown at 158°. Figure 9 shows the typical heat-set curves for a series of samples that contained methylated methylol melamine or urea formaldehyde as the thermosetting resin along with plasticized vinyl chloride. For comparison and to gage the effect on heat-set of systems containing more than one thermoplastic resin, two additional samples were tested. These were treated with a formulation that contained a thermosetting resin and unplasticized styrene butadiene in addition to the plasticized vinyl chloride. To maintain an equal thermoplastic-to-thermosetting-resin-solids ratio

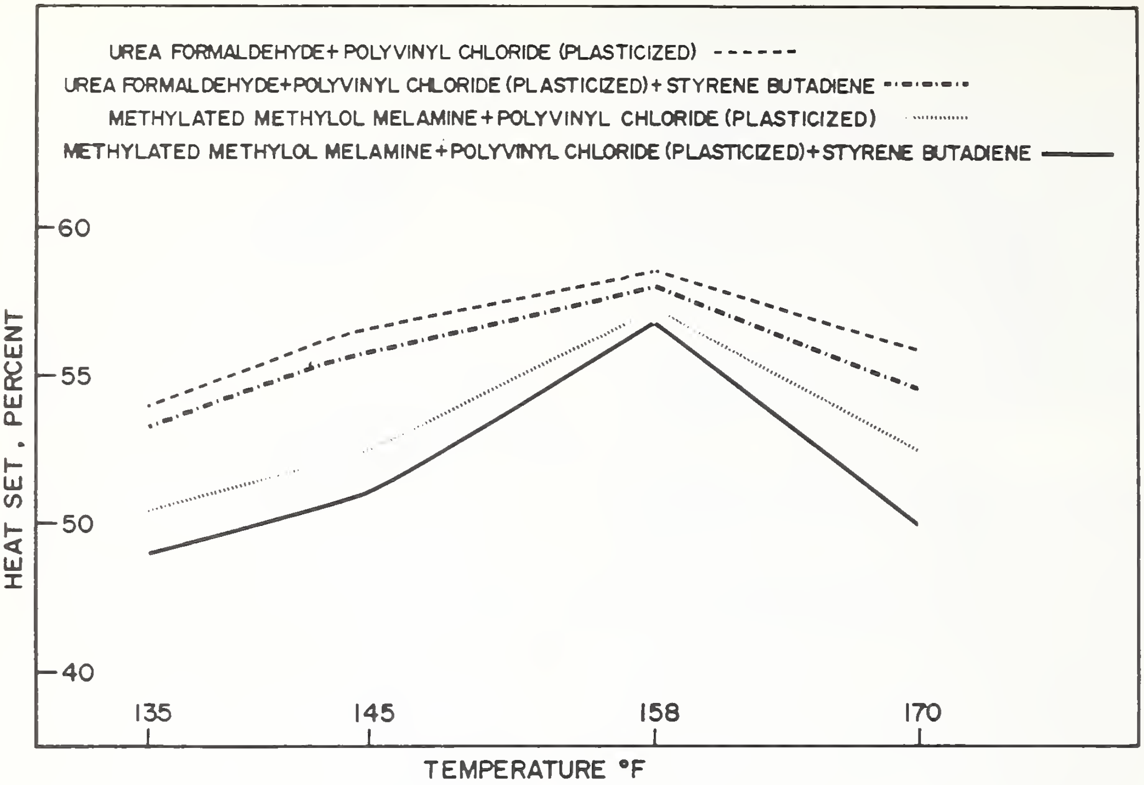

Figure 9.—Affect of internal plasticization of the thermoplast on heat set.

in this case, the vinyl chloride component was reduced to 35 percent and the styrene butadiene content was 15 percent, while the thermosetting resin was held at 50 percent. Figures 7. 8, and 9 corroborate the hypothesis that a second-order process point is present in cotton that has been treated with thermoplastic and thermosetting resins.

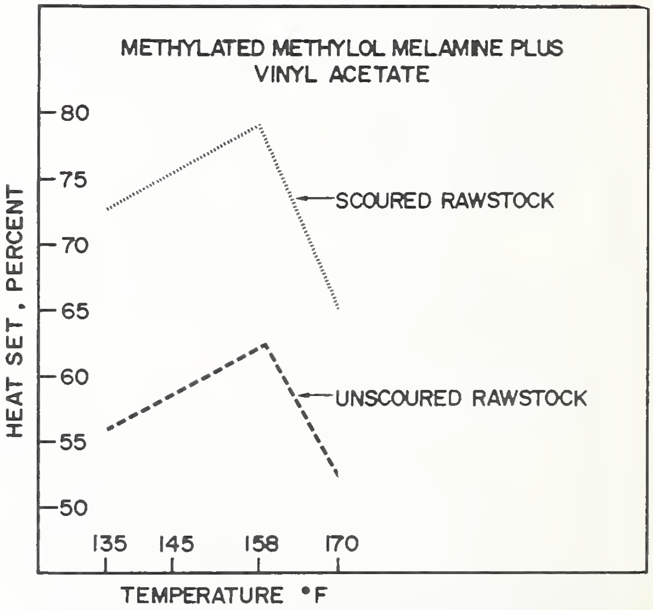

Figure 10.—Effect of scouring on heat set values.

Some possibility existed that where unscoured and unbleached cotton fibers were treated with resins, the impurities on the surface of the cotton might be contributing to the observed heat-set properties. Cotton wax, for example, is generally reported as having a melting point of 168.7° F. (3, 5). Because the wax is not a true single compound, softening or plastic flow might be assumed to occur over a temperature range that could include the 158°, or that the data shown herein were not obtained at the true critical point which could be anywhere between 145° and 170°. Some possibility also exists that one or more of the resins used could react with the wax, even preferentially with a lower melting component of the wax. Figure 10 shows the curves obtained when heat-set values were plotted against temperature for samples of cotton batting that were produced from scoured and bleached rawstock in one instance and from unscoured, unbleached fibers in the other. The treating formulation for each of these was methylated methylol melamine and vinyl acetate in equal solids proportions. The heat-set values were higher in all cases for the sample prepared with scoured rawstock. From these data it can be concluded that cotton wax is not a factor in the observed poor-resilience behavior of resin-treated cotton batting at 158°.

The persistence of the peak in heat-set at 158° F. under all of the above conditions suggests that the cotton itself is contributing significantly to the observed phenomenon. Since cotton is considerably more plastic when wet than dry, it can be postulated that water is, indeed, a good plasticizer for cotton. It can also be postulated that the heat-set performance might in some way be related to the water-sensitive bonds within the cellulose.

It was observed that there is a difference existing in the equilibrium moisture content depending upon the resins used for treatment. Statistical evaluation of the data confirms that the difference is real at the 95-percent confidence interval. It is recognized that the sorption characteristics of cotton fibers depend to a great extent upon the previous history of the fibers (5), and that the effects of adsorbed water are twofold: (a) that which influences physical properties, such as strength, elasticity, and rigidity; and (b) that which maintains vapor pressure and provides the necessary water for chemical reactions (3). An examination of the moisture equilibrium data from figure 11 points to the fact that the moisture content of the samples at 158° F. had fallen to about 1. 7-percent. This level is the moisture that is probably of type (a) and is intimately associated with the molecular structure of the cellulose (17, 19).

It will be noted that there are several changes in the slope of the curve. These occur at about 100° F., 135° and about 158° The first two of these are probably directly associated with drying phenomenon. In the case of the 158°, it might be suspected that the water necessary for plasticization of the cotton has been removed by the 22-hour exposure to this temperature, leaving only the water directly associated with the chemistry of the cellulose structure. On this basis, the tendency of the resin-treated cotton batting to recover from compressive stress at this temperature might be explained as a complex interaction between the resins that inhibit the cotton from rapidly regaining moisture on the one hand, and a repositioning of the bonds between the fibers at points of contact due to plastic flow on the other.

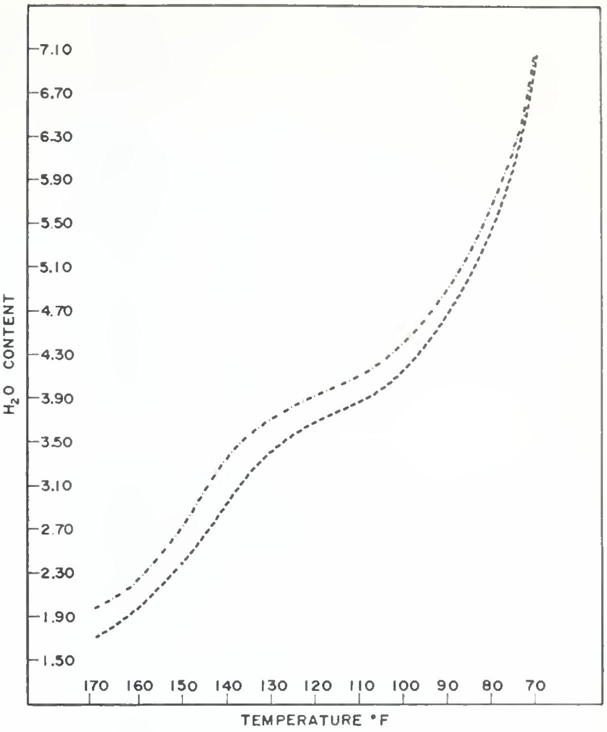

Figure 11.--Equilibrium moisture content after 22 hrs.

To evaluate this possibility, duplicate samples of treated cotton batting were compressed to one-half their original thickness and stored at 158° F. for 22 hours. Upon removal from the oven half of the samples were permitted to recover from this test loading at 80° and 65-percent relative humidity; the other half permitted to recover from the loading at 80° and 85-percent relative humidity. The data derived are shown in table 1. From the table it can be seen that after 30 min. and 120 min. the heat-set of the samples permitted to recover at 65-percent relative humidity were greater than the heat-set values of the samples that recovered at 85 percent relative humidity. This data suggest that at 85-percent relative humidity the absorption of moisture by the cotton is sufficiently fast to overcome, in part, the tendency of the thermoplastic resin to develop energy potential during cooling. Thus a greater elasticity or return to the original configuration is displayed. At the 65-percent relative humidity the combination of thermoplastic and

Table 1. --The effect of humidity upon heat set from sustained deformation loading at 158° F.

Sample no.	Recovery conditions Temp.	Relative humidity	Heat set, percent based on half thickness 0	30	120
	° F.	Percent	Min.	Min.	Min.
A	80	65	72.0	52.3	46.6
A	80	85	72.1	48.6	37.8
B	80	65	76.8	63.6	50.9
B	80	85	74.5	51.0	29.5
C	80	65	77.0	64.0	54.7
C	80	85	78.5	60.7	42.8

thermosetting resins used in the treatment seem to effectively slow down the moisture regain rate to the point where the lattice structure develops energy potential from cooling more rapidly than do the cotton fibers from moisture absorption. At the 65 percent relative humidity a point is reached quite rapidly where the cotton fibers and the thermoset resin bonds within the fibers cannot overcome the energy of the external thermoplastic bonds between fibers. Furthermore, if a second-order process point does exist at 158° F. for the cotton-thermoplastic resin-thermosetting resin system used in this work, it seems quite possible that sufficient plastic flow or deformation could have taken place within the resins that return to the original array would be inhibited by the new configuration of the resin molecules.

A possible explanation for the lower heat-set values observed at 170° F. seems to lie in the fact that water to supply the chemical requirements of the cellulose is absorbed much more rapidly than water associated with the vapor pressure. The absorption of this moisture is accompanied by the release of heat which functions as a plasticizing agent for the thermoplastic resins. Under these conditions the cotton fibers could be expected to gain energy more rapidly, and for a longer time than would occur under the conditions of the 158° F. test. As a consequence, the fibers have a better opportunity to regain a greater proportion of their original configuration in the array.

Conclusions

The elastic performance of cotton fibers that have been treated with thermoplastic and thermosetting resins is adversely affected by prolonged exposure to 158° F. when these fibers are in cotton batting array.

The presence of a peak in the curve obtained when heat set is plotted against temperature suggests that a second-order transition point exists for cotton-thermoplastic resin-thermosetting resin systems at temperatures between 145° F. and 170° with the peak occurring close to 158°.

Plasticization of the resins with alkyl aryl phosphate or dioctyl phthalate at the 5- to 7-percent level based upon thermoplastic resin solids does not eliminate the peak in the heat-set values. Similarly, the use of an epoxy plasticizer does not shift the heat-set values until the amount added is 30-percent or more in terms of thermoplastic resin solids.

Cotton wax is not a significant factor in the behavior noted; in fact, unscoured, unbleached cotton fibers gave products with less heat-set than corresponding products made from scoured and bleached cotton fibers.

The equilibrium moisture content of the cotton fibers seems to play an important part in the ability of thermoplastic and thermosetting resin-treated cotton batting to recover from compressive deformation stress, with the moisture content after 22 hr. at 158° F. being apparently a critical value.

Likewise, the relative humidity during the time of recovery seems to contribute significantly to the ability of cotton batting to recover from compressive deformation with higher relative humidities increasing the amount of recovery measured. These data, if projected to wash-wear or durable-press cottons, point to the possibility that temperatures around 158° F. during processing or subsequent handling of such products might have an adverse effect upon the performance of such products.

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THE BLOCKING OF ACCESSIBLE SITES FOR THE IMPROVEMENT IN PHYSICAL PROPERTIES OF CROSSLINKED COTTON FABRICS

[Summary]

by
T. A. Calamari, A. M. Walker, A. S. Cooper, Jr., and S. P. Schreiber
Cotton Finishes Laboratory
Southern Utilization Research and Development Division

(Presented by T. A. Calamari)

When cotton fabric is treated with certain hydrophobic materials prior to conventional crosslinking, a significant improvement in physical properties over nonpretreated crosslinked fabric is observed. Aqueous solutions of ammonium stearate, zirconyl ammonium carbonate, and a mixture of both reagents have been padded onto cotton fabric, dried, and then thermally decomposed to yield stearic acid, zirconium oxide, or a combination of both hydrophobic agents. It is believed that these agents are deposited at or near those sites on the cellulose lattice where initial crosslinking normally takes place. Presumably, subsequent reaction with crosslinking agents is then reduced at these normally preferred sites because of hydrophobic blocking. This makes less accessible reactive regions participate in crosslinking. Regardless of whether or not the sequence of reactions takes place as is proposed, the physical result of such a hydrophobic pretreatment is that relatively good smooth-drying properties and wrinkle-recovery values are obtained without the excessively large decreases in breaking strength, tearing strength, and abrasion-resistant values which normally accompany a conventional crosslinking procedure.

Before crosslinking, ammonium stearate pretreated fabric exhibits an exceptionally high Stoll flex life (in some cases, seven times that of an untreated control). This increase in flex life is not totally unexpected, because fiber lubricant properties of stearic acid are well known; however, the degree of retention of flex life after crosslinking is more pronounced than has been observed with nonhydrophobic fiber lubricants. The breaking strength and tearing strength values for crosslinked ammonium stearate pretreated fabric are significantly higher than those of unpretreated crosslinked controls. Results from wash, tumble-dry, wear tests on sample cuffs indicate a significant improvement in resistance to wear over conventionally treated cuffs.

The uncrosslinked zirconyl ammonium carbonate pretreated fabric exhibits a large reduction in flex life when compared to an untreated control. It is presumed that when the zirconyl ammonium carbonate is heated, the zirconium oxide produced is in the form of small abrasive particles which act as a pomice to reduce the flex life of the fabric. Upon subsequent crosslinking, a large increase in flex life is observed. This is quite unusual, and it can only be assumed that the particles of zirconium oxide are in some manner encapsulated or encaged by the crosslinking formulation. This encaging of zirconium oxide does not seem to prevent its hydrophobic redirecting of crosslinks to less accessible sites, however, because the pretreated crosslinked fabric is much more resistant to abrasion and has significantly higher values for both breaking strength and tearing strength than conventionally treated cloth.

A combined pretreatment incorporating both ammonium stearate and zirconyl ammonium carbonate in one pretreating solution appears to be more effective than either reagent used separately. The precise concentration of each component has not been fully determined as yet, but preliminary results indicate an enhanced effectiveness for such a pretreatment.

While the pretreated crosslinked products obtained thus far have not possessed wrinkle-recovery values sufficiently high to be of value for durable-press fabric, they are excellent for use in high performance wash-wear applications. If one attempts to increase the wrinkle recovery angle of the pretreated fabric beyond 270° or 275° (W+F) by increasing the extent of crosslinking, he reduces the beneficial effect of the hydrophobic pretreatment.

DISCUSSION

Question: Is it true that the conversion of ammonium stearate to stearic acid was not complete and that stearamide was probably formed as a byproduct?

T. A. Calamari: No, unless the thermal decomposition was carried out under ammonia gas and under pressure, stearic acid was the only product.

Question: If flex and other abrasion tests of treated fabrics are made under identical performance levels, i. e., about the same WRA-W+F, are the results of such tests completely meaningful?

T. A. Calamari: All of the data did not reflect the results of identical performance levels, but much of it did.

Question: Since ammonium stearate tends to form relatively large miscella in aqueous solutions, it is possible that it does not penetrate the cotton fiber. Couldn't the increase in flex life and resistance to abrasion be attributed to a lubrication of the outer fiber wall?

T. A. Calamari: In the case of ammonium stearate, this is a good possibility.

Question: Would you clarify how zirconium oxide at first reduced the flex life and then upon crosslinking extended it?

T. A. Calamari: Probably an encapsulating or encaging phenomena is the most likely explanation.

FILLING COMPACTION OF FABRICS

[Summary]

by
W. G. Sloan, M. J. Hoffman, A. M. Walker, and A. S. Cooper, Jr.
Cotton Finishes Laboratory
Southern Utilization Research and Development Division

(Presented by W. G. Sloan)

A proposed method of mechanically compacting fabrics in the filling direction and some preliminary work that has been done are described.

Fabrics can be compacted in the warp direction by several methods. It has been shown that warp compaction improves such properties as warp stretch, warp and filling flex abrasion resistance, and flat abrasion resistance.

Fabrics are also being slack mercerized commercially to produce filling stretch which is filling compaction by chemical means.

The purpose of this research is to determine how mechanical compaction in the filling direction can best be accomplished and to what extent it improves fabric properties.

Filling compaction can be achieved by running wet fabric in contact with a series of rubber covered bow rolls. The fabric contacts each bow roll at the peak of the bow and maintains contact for about 180°, or halfway around the roll, leaving the roll at the valley. Since the wet fabric conforms to the rubber surface of the rolls it becomes narrower as it passes from the larger outside arc at the peak of the roll to the smaller inside arc at the valley of the roll.

A schematic drawing of a proposed filling compaction unit is shown in figure 1. This is an overhead view of the unit. The fabric first passes over a guide roll and then alternately passes under and over a series of six bow rolls contacting each bow roll at the peak of the bow and leaving it at the valley. The bow rolls are set so as to be in contact with each other. This keeps the fabric in constant contact with the bow rolls until it leaves the last bow roll and should prevent wrinkles from forming until it leaves the last compacting bow roll.

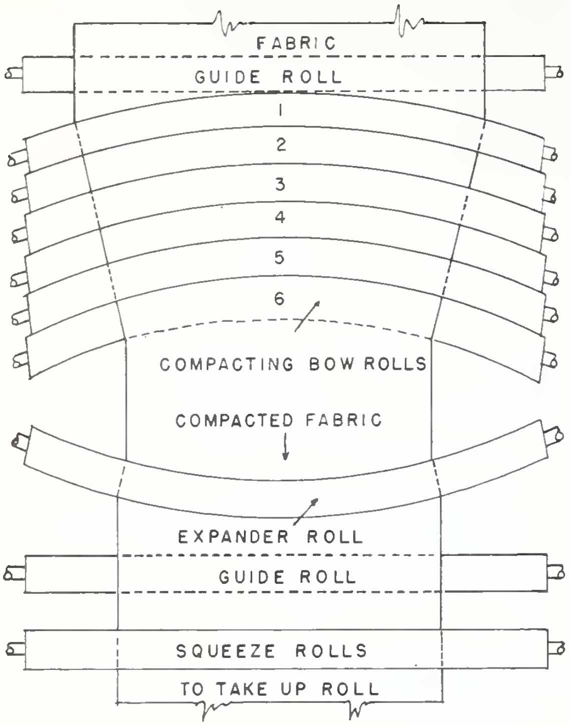

Figure 1.–Plan View of Filling Compaction Unit.

After passing over the last compacting bow roll the fabric passes under an expander roll to remove any wrinkles that may be present. This expander roll is the same type or roll as the compacting bow rolls, but is placed with the peak of the bow set in a different position so that when the fabric contacts this roll it is widened slightly. The fabric then passes over a guide roll and between two squeeze rolls to a batcher or take-up mechanism.

The six compacting bow rolls have different amounts of curvature. The first bow roll that the cloth contacts has the least amount of curvature; each succeeding roll has more curvature than the previous roll as the outside arc of each bow roll is the same as the inside arc of the preceding roll.

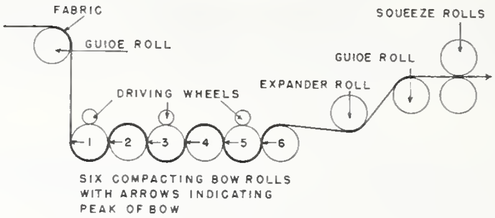

Figure 2.–Sectional view of filling compaction unit.

A sectional view of the proposed compacting unit is shown in figure 2. The position of the peak of the bow in the six compacting bow rolls is at 9 o'clock as indicated by the arrows shown with each roll. The other arrows in the figure are for identification purposes only. The peak of the bow in the expander roll is set at about 2 o'clock. Figure 2 also shows driving wheels driving compacting bow rolls 1, 3, and 5 which the fabric passes under. These driving wheels may not be required, but can be used if the takeup mechanism does not exert sufficient lengthwise tension on the fabric to turn the bow rolls or if any slippage occurs. Although six compacting bow rolls are shown in this unit, the number of compacting rolls could vary with processing requirements, size, and curvature of the rolls, and other factors.

In preliminary experiments high lengthwise tension has been used to mechanically compact fabrics a small amount during resin treatment. The results of physical property evaluation tests were encouraging.

An experimental filling compaction unit has been built which contained three bow rolls that were not in contact with each other. Fabrics run on this unit were compacted a small amount, but wrinkling occurred between the bow rolls. The unit is being modified with the bow rolls set as closely together as possible to minimize wrinkling. There is every indication that this modification will permit compacting of fabrics for evaluation.

DISCUSSION

Question: Does the compacting device discussed have a tendency to put bow into the compacted fabric?

W. G. Sloan: Insufficient work has been done to comment on this aspect of the process. The major problem encountered thus far has been the wrinkles produced during compaction.

Question: Is filling compaction being obtained at the expense of warp elongation?

W. G. Sloan: The tension experienced by the fabric during compaction is not much greater than that normally encountered during textile processing; therefore, I do not feel that this is a problem.

THE INFLUENCE OF SPECIAL FINISHING TECHNIQUES ON DURABLE PRESS PERFORMANCE

[Summary]

by
R. J. Harper, Jr., E. J. Blanchard, and G. A. Gautreaux
Cotton Finishes Laboratory
Southern Utilization Research and Development Division

(Presented by R. J. Harper, Jr.)

Cotton durable-press fabric with 30-percent or more warp elongation was produced by padding the fabric with a finishing formulation, drying; warp compacting the sensitized fabric and then curing the compacted fabric. This unusual elongation had a pronounced effect on fabric abrasion performance both as measured by standard textile tests and by laundry tests using simulated trouser cuffs.

A cotton oxford (4.1 oz./sq. yd.) and a cotton denim (6.6 oz./sq. yd.) were used. These fabrics were given a two-step combination treatment using a polymer and a crosslinking agent. The polymer used was a polyurethane, (Wyandotte E-406A) while the crosslinking agent was a commercial dimethylol dihydroxyethyleneurea. The basic treatment sequence has been published elsewhere[1] The treatment consists of a pad-dry-pad-dry sequence in which the polymer is applied in the first bath and the crosslinking agent is applied in the second bath. The oxford cloth was padded with 15-percent polyurethane (Wyandotte E-406A), dried, then padded with 7.2-percent dimethylol dihydroxyethyleneurea (Permafresh 183), catalyst X-4 (a modified zinc nitrate) and 2-percent polyethylene. The denim was treated with 16-percent polyurethane E-406A, dried, then padded with 6.3-percent dimethylol dihydroxyethyleneurea (Permafresh 183K), catalyst X-4, and 1-percent polyethylene. The sensitized fabrics were then shipped to the Fabric Research Laboratory, where they were given varying degrees of compaction by means of a warp compactor. The sensitized compacted and the noncompacted control were then cured and subjected to various tests.

The immediate effect of compacting is a progressive increase in filling thread count with increasing degree of compaction. The control oxford had a filling thread count of 52 and the compacting filling ranged from 56 to 70 threads per inch. The compacted denim had a filling thread count of 67 to 69 compared with 58 threads per inch for the control. The compacted oxford had conditioned wrinkle recovery values as high as 333° (W+F), compared to 316° (W+F) for the noncompacted control. The warp and filling breaking strengths of a highly compacted oxford were 32.8 and 39.5 lb. compared to corresponding values of 36.6 and 30.1 lb. for the noncompacted control. The increased filling breaking strengths can be related to the higher filling thread count. The different amount of compaction elongation given individual warp ends in the short strips used for tensile testing may be a major factor in the reduced warp breaking strengths.

While the control oxford had a warp elongation at break of 3.7-percent, the compacted oxford had an elongation-at-break ranging from 10 to 35-percent depending upon the degree of compaction. The filling elongation to break was slightly improved over the control.

While both the warp and filling tearing strengths were improved by compacting, the filling tearing strength was improved to a lesser extent. The increase in filling tearing strength is somewhat remarkable considering the increase in filling thread count. A highly compacted oxford had a warp tearing strength of 1,383 g. The comparable values for the control were 1,000 g. in the warp and 967 g. in the filling. Pressing the compacted fabric before curing generally reduces the tearing strength somewhat but values are still significantly improved over the control. Compacting reduced fabric stiffness significantly as indicated by reduced warp bending moment.

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[1] Blanchard, Eugene J., Harper, Robert J., Jr., Gautreaux, Gloria A., and Reid, J. David. Urethanes Improve All-Cotton DP. Textile Indus. 131(1): 116–119, 122, 143. 1967.
Harper, Robert J., Blanchard, Eugene J., Lofton, John T., and Gautreaux, Gloria A. Stiffness, An Important Factor in the Abrasion Performance of Durable-Press Cottons. Textile Res. J. 37: 233–241. 1967.

Analogous trends in breaking tearing strengths and elongation-at-break were also observed for the heavier denim fabric.

With respect to abrasion testing, however, a definite difference between the oxford and denim was observed. With the oxford, the compacted fabric had lower Stoll flex values than the noncompacted control. A reduction in the weights used with the Stoll Flex machine from 4 lb. to 2 lb. to 1 lb. increased the number of cycles to failure by a fivefold factor on each occasion. However, in all cases, the relative ratio between the compacted and control was maintained. Quite a different set of results was observed with the denim. Here, the compacted fabric had twice the number of cycles as the control. Furthermore, the denim sample with the highest elongation had a flex value approximately three times the control and approximately 25 times untreated cotton. The reason for the difference between the two fabrics is believed to be that the operator removes most of the easy stretch in inserting the lightweight oxford in the Stoll tester. With the heavier fabric some of this stretch is left in, so improved flex performance is observed. With the Accelerotor edge test, the compacted fabrics showed improved performance over the controls.

In laundry testing, using durable-press cuffs, we also observed improved performance for the compacted samples. Thus, with the oxford fabric, the noncompacted control fabric with a combination treatment had minor damage after eight wash cycles. The average of six compacted samples had minor damage after 22 cycles. With the denim, a standard crosslinked control had major damage after four laundry cycles. The combination control (noncompacted) had major damage after 12 laundry cycles. The denim cuffs prepared from compacted fabrics lasted 15, 18, and 21 cycles before major damage occurred. The number of laundry cycles increased with the degree of compaction in the fabric.

Based on laundry cycles, the abrasion performance was from 150 to 300-percent of the control with the oxford fabric and 125 to 175-percent for the denim fabric with the combination treatments. The somewhat lower relative improvement achieved with the denim fabric than with the oxford probably resulted from two factors. First, the warp elongation-at-break of the control denim was more than twice that of the oxford control. Second, the compacted denim was significantly heavier than the control denim. An increase in fabric weight tends to accelerate cuff tip wear. With a control denim closer to the weight of the compacted denim, a better relative performance would have been achieved.

The increased elongation-at-break stretch, toughness, reduced fabric stiffness, and the suppleness of the compacted fabrics were contributing factors to the improved abrasion performance achieved with the compacted samples. Extension of this type treatment to other finishes will be reported later.

ACKNOWLEDGMENT

We thank Thomas Constantine of the Fabric Research Laboratories for compacting our test fabrics with the Fabric Research Laboratory Warp Compactor.

FOURTH SESSION: L. H. Hance, Fieldcrest Mills, Inc., Chairman

VARIATIONS OF THE WET FIX PROCESS

[Summary]

by
S. L. Vail, A. H. P. Young, G. B. Verburg, J. D. Reid, and W. A. Reeves
Cotton Finishes Laboratory
Southern Utilization Research and Development Division

(Presented by S. L. Vail)

The wet fixation process, as described in previous presentations and publications, involves the fixation of a polymer-former and a crosslinker to cotton fabric under aqueous, acidic conditions. In this process the wet fabric is heated in a sealed container or allowed to stand overnight. The resins are firmly fixed in the cotton and the fabric, after rinsing and applying catalysts and softener, can be used in the production of durable-press garments.

This process was first announced in early 1966 by the National Cotton Council of America and the laboratory procedures were described in detail. The strong points of the process begin with the production of an essentially odorless, sensitized fabric for durable press. In addition, the cured fabrics are smooth-drying with significantly less impairment of tearing strength and Stoll flex abrasion than conventionally treated fabrics with equivalent smooth-drying properties. The present study has attempted to improve the process in a practical sense and to examine further the properties of the finished fabric.

The first or fixation step appears to be the most important phase of the process. The agents are fixed, but they must be fixed in such a manner that the subsequent curing operation produces the desired properties. High add-on does not assure the attainment of these properties. It appears that the degree of swelling and the manner of partial polymerization at the time of fixation have a major influence on the success or failure of a certain treatment. Because of this importance, fixation under varying conditions has received considerable attention. Compared to the conventional fixation in a sealed container, the following conditions are considered to be improvements: (1) Slight or no pre-drying followed by standing overnight at room temperature, and (2) use of a swelling agent (tetraglyme) which allows the fixation to be carried out during the time it takes to heat the wet fabric in an oven and dry it to a moist condition. Procedures which are presently considered failures include: (1) Pre-polymerization of the resin mixture followed by drying and (2) fixation under steam pressure. However, more work remains to be done on the latter process. Finally, the use of inorganic salts (sodium nitrate or sodium chloride) as swelling agents during fixation (normal drying) is being studied. Results from these studies are incomplete, but early treatments produced cured fabrics of relatively low crease recovery.

Testing of the cured fabrics for abrasion resistance remains a problem. The Stoll flex test has been used to eliminate poor treatments, but results from this test are extremely misleading when compared to the results from wash-dry tests using cuffs. For this reason the conclusions given in this summary with regard to improvements are based largely on the results of tests on cuffs subjected to 30 wash-dry cycles.

One-component systems do not produce as good a result as the combination of a melamine resin (Aerotex 23-S or M-3) with a crosslinker (Permafresh 183, urea-formaldehyde, methylol carbamates, or formaldehyde) does. A highly methylated methylol melamine and urea-formaldehyde have also been tried as polymer formers, but results to date have been discouraging. Use of other resins which were not melamine derivatives have been tried as polymer formers, but they did not appear to be chemically suited for the process. Various carbamates and Permafresh 183 have been used much more than the other crosslinkers. Methylolmelamines in a one-component system do not appear satisfactory, but the addition of formaldehyde produces an improvement based on Stoll flex data. This use of formaldehyde is interesting from an economical and also from a theoretical standpoint.

Cured fabrics from the wet-fix process were found to possess moisture regains equal to that of cotton or up to 10 percent higher. Also, the finished fabrics accepted a direct dye (Solatine Blue) to a slight extent suggesting that the fabric is not highly crosslinked, nor is the fabric relatively swollen when crosslinking occurs. A very mild acidic hydrolysis will not lower the crease recovery, but both the tearing strength and Stoll flex values are improved slightly. Stronger hydrolysis (pH = 1, 40°, 30 min.) reduces the crease recovery of these fabrics more than the same hydrolysis conditions reduce the crease recovery of fabrics treated in a conventional manner with the same agents in a one-component system. In other words, finishes of wet-fix melamine-carbamate treated fabrics hydrolyze faster than conventionally treated fabrics crosslinked with melamine or carbamate resins. Curing conditions are known to have a slight effect on the stability of crosslinks. This may be the major cause for the hydrolysis rather than formation of a chemically susceptible crosslink.

Polyurethane and softeners or both are useful for improving the appearance of wet-fix fabrics. Use of these auxiliaries can, however, render the Stoll flex test completely useless for predicting the life of a cuff in wash-dry cycles.

Curing of wet-fixed fabrics have been carried out successfully using zinc nitrate or magnesium chloride. However, in our experiments we found that care had to be exercised during curing or a tender fabric resulted. In most cases, particularly in our variations of this process, it was better to use magnesium chloride. Similarly, in our variations it was generally better to limit the add-on to 4 to 8 percent and to make use of the auxiliaries for achieving sharper creases and higher crease recovery angles. Achievement of smooth-drying properties was rarely a problem.

In summary, the methods of wet fixation have been adapted to the production of fabrics in a practical manner with considerable improvement in Stoll flex tests. However, the Stoll flex test can be used at best only to eliminate failures. The use of cuffs in a wash test has equated many promising treatments to the controls or lessened the value of a promising treatment. The greatest improvement is noted for cuffs or medium creases which have been fixed in the presence of tetraglyme. Auxiliaries are used with this treatment to improve the appearance of the cuff. If a sharp crease is required, the improvements we have obtained, using the standard wet fix or our variations, are generally not enough to carry the cuff through 30 washes without the appearance of a hole. Thus, the fabrics from this process appear at the present state of development to be best suited for use in garments where smooth-drying properties are more important than sharp creases.

DISCUSSION

Question: At what temperature was the fabric cured? What is the effect of overcuring?

S. L. Vail: Most fabrics were cured at 160° C. for 3 min. Overcuring is detrimental--a cure period of five minutes was safe, but 7 min. often produced a tender fabric with little or no gain in appearance over the milder cured fabric.

Question: What is the effect of shelf storage?

S. L. Vail: We have done no storage experiments.

Question: What did you do with tetraglyme?

S. L. Vail: Twenty percent tetraglyme was padded on the fabric along with the polymer former and the crosslinker. The fabric was dried at 60° C. for three min. at pH_2. The "dried fabric" was still moist. It was taken out and washed immediately.

Question: Did you get results that were comparable to the best results obtained using a conventional treatment?

S. L. Vail: Essentially yes, but performance was slightly lower. We get reasonably good cuffs. It is very difficult to match treatments, especially when using cuffs.

Question: What are the relative merits of wet fix and polyset?

S. L. Vail: No comparison was made due to different fabric constructions that were used in the two treatments.

Question: On the fourth slide, was the wash-wear rating obtained by the overhead method?

S. L. Vail: No, side-angle lighting was used.

Question: Do you realize the melamine resin is subject to Cl damage?

S. L. Vail: Yes. We would expect yellowing during multiple washes, however, we would not expect strength losses due to chlorine damage.

THE USE OF REACTIVE SOFTENERS AND REACTIVE ADDITIVES IN DURABLE PRESS FINISHING

[Summary]

by
E. J. Blanchard, R. J. Harper, Jr., J. S. Bruno, and J. D. Reid
Cotton Finishes Laboratory
Southern Utilization Research and Development Division

(Presented by E. J. Blanchard)

A number of reactive softeners and additives were investigated to determine their effectiveness in improving the abrasion resistance of crosslinked, durable-press, cotton fabric. Long-chain agents investigated included polyethylene and polypropylene glycols, long-chain alkyl polyethylene oxide alcohols, octadecyl ethylene urea, octadecyl isocyanate, and a hydroxymethyl derivative of a condensation product of a higher fatty-acid chloride with polyethylene polyamide, Sapamine WP. Other agents investigated included long-chain dialkyl dimethyl ammonium salts, and a long-chain softening agent containing a positive charge, Viva Fibreactive.

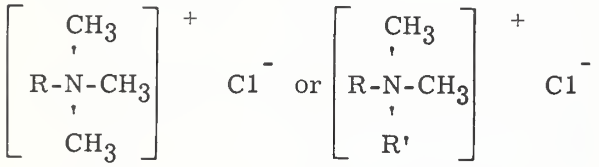
Of the reactive softeners and additives investigated, those which improved resistance to abrasion were agents that contained a positive charge.

The polyethylene and polypropylene glycols used were water-soluble polymers with good lubricating properties having a molecular weight range from 300 to 20,000. Although fabric properties, such as tearing strength or breaking strength, could be improved with these polymers, there was only slight evidence of a positive contribution to abrasion resistance. This was shown by using both laboratory test methods and laundry wash, tumble-dry cycles with trouser cuffs. It appears that these glycol agents are not useful in imparting improved abrasion resistance to cotton fabrics. Also studied were long-chain alkyl polyethylene oxide alcohols, in which the alkyl groups ranged from 10 to 20 carbon atoms and in which the ethylene oxide units comprised 40 to 60-percent of the total molecular weight. These imparted some lubricity to the fabric, as indicated by improved tearing strength. However, there was only a slight improvement in performance of durable-press cuffs as shown by laundry tests. Khaki twill fabric treated with octadecyl ethyleneurea in combination with a crosslinking agent showed improvement in the number of laundry cycles to major damage. With 2-percent of this softening agent there was a 40-percent increase in the number of wash cycles to major damage compared with the crosslinked control sample. At the 5-percent level the number of wash cycles to major damage increased 100-percent over a control sample with a similar level of wrinkle recovery performance.

Octadecyl isocyanate was found to be effective in increasing the number of cycles to major damage approximately 100-percent when applied to cotton fabric from an emulsion followed by crosslinking agent. Although the samples had satisfactory wash-wear ratings and bound formaldehyde was equivalent to the control, the measured wrinkle recovery angles were 50 to 60° lower than the crosslinked control. The level of wrinkle recovery performance was increased by applying the isocyanate from organic solvent; however, the wrinkle recovery angle in this case was still some 20° lower than a crosslinked control. This lower angle may account, in part, for a 50-percent increase in the number of wash cycles to major damage over that of the control sample. However, samples treated with octadecyl isocyanate have the advantage of having a soft hand that enhances the wash-wear rating in some cases.

A hydroxymethyl derivative of a condensation product of a higher fatty-acid chloride with polyethylene polyamine (Sapamine WP) proved effective in extending the number of wash cycles before major damage occurred to khaki trouser cuffs. A twofold increase in the number of wash cycles over that of the crosslinked control was obtained while still retaining a wrinkle recovery angle of 263° compared to 268°. This material is readily dispersible in water and can be used in a single pad-bath formulation with crosslinking agent. Laboratory tests on lightweight fabric, however, did not show any improvement over the crosslinked control in a comparison of breaking and tear strengths, and Accelerotor and flex abrasion tests. Also, there was no contribution to improvement of wrinkle recovery with the light fabrics.

Long-chain quaternary ammonium salts have been found to increase the number of laundry cycles before major damage occurred in trouser cuffs. These compounds have the following general formula,



$$\left[\begin{array}{c} CH_3 \\ | \\ R\text{-}N\text{-}CH_3 \\ | \\ CH_3 \end{array}\right]^+ Cl^- \quad \text{or} \quad \left[\begin{array}{c} CH_3 \\ | \\ R\text{-}N\text{-}CH_3 \\ | \\ R' \end{array}\right]^+ Cl^-$$

where R and R' are long-chain alkyl groups.

The most promising compound of this type found to date is the dialkyl dimethylammonium chloride in which the alkyl groups are composed of octadecyl and hexadecyl units in a 3 to 1 ratio.

Quaternary compunds of this type are cationic surface active agents. Because of the charge, they are substantive and become attached to the negative cellulosic sites through the formation of an electrochemical bond with the nitrogen. Presumably, the hydrocarbon chains do not form a continuous film but are free to move. This accounts for the mild lubrication conferred on the fabric by these groups. In fabric treatments it is necessary to use a two-step process, because the quaternary compounds are not compatible with the metal-salt catalyst used in the resin formulation.

Another difficulty, which arises with the use of the quaternary compounds, concerns the concentration level that can be used. In general, higher concentrations of the quaternary were accompanied by a decrease in the wrinkle recovery angle over that obtained with lower concentrations. In most cases a suitable degree of wrinkle recovery was obtained only if the fabric was rinsed with 5-percent acetic acid before applying the crosslinking agent. Furthermore, at higher concentration levels, the fabric is somewhat resistant to wetting and this may reduce the wet pickup of the crosslinking resin formulation.

On 7 oz. khaki twill fabric, octadecyl hexadecyl dimethylammonium chloride was instrumental in increasing the number of cycles to major damage over 100-percent. Wrinkle recovery angle after the 30th wash was 275° compared to 280° for the control. When a similar type treatment was used in conjunction with a polyacrylate for additional protection of the fabric, the performance level was similar to that obtained with the addition of the polyacrylate. The crosslinked control treated with polyacrylate showed the usual increase in wrinkle recovery angle in addition to a slight improvement in the number of laundry cycles to major damage.

Diphenylstearyl quaternary ammonium chloride was also effective in increasing the number of laundry cycles to major damage over that obtained with a crosslinked control. A sample treated with 5-percent of the quaternary lasted 22 cycles before receiving major damage compared with the crosslinked control which lasted only four cycles. Although the quaternary treated sample had a wrinkle recovery angle of 261° compared to 288° for the crosslinked control; a wash-wear rating panel gave the quaternary treated sample a higher rating than the control.

A long-chain softening agent containing a positive charge, Viva Fibreactive, was responsible for allowing trouser cuffs to go through 30 laundry cycles without major damage. This material is compatible with crosslinking resin formulations, better results were obtained by using a two-step process in which the Viva Fibreactive was applied initially, since wrinkle recovery was suppressed with a single pad-bath formulation. Concentration levels of the pad-bath solutions were 1- and 3-percent. On denim both 1- and 3-percent Viva Fibreactive treated cuffs went through 30-laundry cycles without major damage. These results were obtained on both mercerized and unmercerized fabrics; however, at the 3-percent level the wash-wear rating on mercerized fabric was lower than that obtained at the 1-percent level. Laundry cycles to major damage of the corresponding controls were 18 cycles on the mercerized denim and four cycles on the unmercerized fabric. The wash-wear rating of the control cuffs was only slightly higher. Therefore, it is possible to obtain good abrasion resistance and wash-wear performance if concentration and method of application are taken into consideration.

DISCUSSION

Question: I have a suggestion for future study with these reactive type softening agents. Dioctadecyl quaternary ammonium chloride is the most commonly used laundry softener. I suggest that a study be made of the improvements which can be obtained by the regular use of such laundry softeners in the washing of cotton durable-press (DP) garments. If the results are favorable, a suggestion for their use could be included on the tags attached to the garments. Their use may aid the utilization of cotton DP garments.

E. J. Blanchard: Good idea. It is planned to do this.

Question: Has the advantage of pre- and post-treatment use of these reactive softening agents been compared? The effects may be quite different.

E. J. Blanchard: This is a good point. To date all applications have been as pre-treatments or as combined treatments with the crosslinking agent.

Question: Have you tried Viva without polyethylene?

E. J. Blanchard: Yes. Although there have been reports that Viva may not be as effective with polyethylene, we did not see any significant difference. However, the crosslinked control included polyethylene and this was used in the slides for easy comparison.

Question: What was the effect of nonionic softeners?

E. J. Blanchard: They were less successful for improved abrasion resistance.

Question: Laundry softeners are usually used at a concentration of about 0.1 percent based on the weight of cloth. Why did you use such high concentrations?

E. J. Blanchard: We have used other concentrations, but those reported here seem to be the best under the conditions employed.

Question: Is the octadecyl ethyleneurea a commercial product?

E. J. Blanchard: Yes.

THE POLY-SET DURABLE PRESS FINISH: RECENT DEVELOPMENTS

[Summary]

by
C. Hamalainen, H. H. St. Mard, W. A. Reeves, and A. S. Cooper, Jr.
Cotton Finishes Laboratory
Southern Utilization Research and Development Division

(Presented by A. S. Cooper, Jr.)

The poly-set process for durable press involves two steps of consecutive pad-dry-cure treatments. In the first step methylol crosslinking agents are deposited on and within the cotton fibers primarily by polymer formation, using weak acid polymerization catalysts. Step two uses a strong latent acid catalyst to cause more extensive covalent crosslinking of the cellulose molecules to obtain the desired smooth-drying performance. The process is suitable for both precuring and postcuring operations. By essentially eliminating the release of formaldehyde after step one, it permits the use of most types of methylol crosslinking agents in a delayed cure process.

A number of metallic acetates have been used as polymerization catalysts in step one. Some of these, such as lead, copper, and manganese acetates, are not suitable because of discoloration of the fabric. The catalysts that have been used more extensively are zinc acetate and zirconyl acetate in acetic acid solution. On the basis of chemical costs and efficiency of polymer deposition, zinc acetate is preferred. However, under some conditions zirconyl acetate may give superior results. The efficiency of resin deposition is generally about 60 percent. Methylated methylol melamine (MMM) is deposited with slightly higher efficiency than dimethylol dihydroxyethylene urea (DMDHEU) with both the zinc and zirconyl acetates. The use of zinc acetate with or without added acetic acid results in fabrics that have higher wrinkle recovery angles after step one and a slightly stiffer hand than when zirconyl acetate is used. This may be a disadvantage in sharp crease formation in a delayed cure, step two treatment. The high wrinkle recovery angles after step one using zinc acetate indicate that some crosslinking occurs in step one. This is supported by cuene solubilities and lamellae separation by swelling and methacrylate deposition.

Crosslinking agents representing most types of n-methylol resins have been studied with emphasis on MMM and DMDHEU singly and in combinations. The resin deposited in step one is durable to process washing for removal of unreacted resin and byproducts. The washed fabric can be sensitized with the latent acid catalyst, dried, and stored in excess of 6 months without formaldehyde release or reduction in the efficiency of crosslinking during step two.

Conventional latent acid catalysts have been used in step two with emphasis on zinc nitrate and magnesium chloride. Zinc nitrate is preferred on the basis of physical properties obtained. The addition of softening agents to the catalyst enhances the physical properties, as well as smooth-drying performance and wrinkle recovery angles of treated fabrics. The use of softening agents in both step one and step two causes further enhancement of the fabric's performance characteristics. In comparison with conventionally crosslinked fabrics, the poly-set process produces fabrics with higher flex abrasion resistance, good tearing and breaking strength, and better resistance to wear as determined by the Accelerotor edge wear test.

The addition of thermoplastic type polymers to step one has been found to cause unusually large increases in the wrinkle recovery angles of step one treated fabrics. Fabrics with up to 300° conditioned and wet wrinkle recovery angles have been obtained without the step two curing step. Since the physical properties of the fabrics are not reduced by the high temperature latent acid cure of step two, much improved physical properties are obtained. For some uses the smooth-drying performance may be adequate for use as a precured finish.

Because the poly-set process involves resin deposition and subsequent crosslinking, possibly by a mechanism different from conventional crosslinking treatments, the resistance to chlorine scorching damage may be adversely affected. Resistance to chlorine damage after step one and step two has been determined with several crosslinking resins. With the exception of DMDHEU, all of the resins showed excellent resistance to chlorine damage after step two. When DMDHEU is used in combination with MMM, excellent resistance to chlorine damage is obtained. The use of zinc acetate as the polymerization catalyst produces fabrics with the highest resistance to chlorine damage.

DISCUSSION

Question: Would you identify polyether, urethane, and acrylic additives?

A. S. Cooper, Jr.: Polyether is Interchem 67; urethane is Wyandotte E-406 A; acrylic is Rohm & Haas K-87.

Question: What is the abrasion after five or more launderings? Are superficial chemicals on surface causing properties?

A. S. Cooper, Jr.: Laundry tests have not been made.

Question: What are the results of cuff wear?

A. S. Cooper, Jr.: Properties of cuffs made on previous occasions lasted well.

Question: How about the high WRA after step one? What is the crease sharpness?

A. S. Cooper, Jr.: Cuffs are made and creased before the final step two cure.

Question: What about chlorine loss on use of zirconium as catalyst?

A. S. Cooper, Jr.: With selected resins the use of zirconium as catalyst is acceptable.

MICROSCOPICAL ABRASION PATTERNS IN DURABLE-PRESS COTTONS

[Summary]

by
M. L. Rollins, I. V. deGruy, T. P. Hensarling, and J. H. Carra
Cotton Physical Properties Laboratory
Southern Utilization Research and Development Division

(Presented by M. L. Rollins)

In the highly successful durable-press treatments now in commercial production, the outstanding drawback is the poor abrasion resistance that invariably accompanies the achievement of crease recovery angles in the region 270° to 300°. The drawback is associated with losses in tensile strength and in elongation. Much work has been concentrated on methods of evaluating abrasion characteristics of cotton treated in the various ways recommended for durable-press. Abrasion test methods have been under close scrutiny, but to date no fully satisfactory method for laboratory testing has been devised. Nevertheless, it has become apparent from all of the testing done that, in general, fabrics with satisfactory smooth-drying properties and satisfactory permanent crease retention have low tensile strengths, low elongations, and low resistance to abrasive failure.

Previous published work involving microscopical observations of fiber breakdown is scanty. Yet information on the mechanism of fiber failure in fabric abrasion is of vital importance to the utilization of cotton in apparel and military goods because of the competition of fabrics of other than cotton composition. The objective of this investigation was to observe microscopically the pattern of fracture in the abrasion of cotton fabrics treated by various promising durable-press procedures to determine differences in the wear pattern associated with the treatment, the properties of the fabric, and the conditions of initial treatment, of subsequent wear or of laundering. Included in these observations are the effects of abrasion in the Accelerotor, the effects of flex abrasion in the Stoll abrader, and the effects of washing and tumble drying in a household type washing machine, as shown in electron micrographs of replicas of damaged fiber surfaces.

Treatments compared were: (1) The conventional pad-dry-cure crosslinking treatment with DMEU and a magnesium catalyst; (2) a laboratory treatment simulating the commercial delayed cure process known as "Koratron"; (3) a laboratory treatment simulating the "wet-fixation" treatment developed at Harris Research Laboratories under contract to the National Cotton Council of America; (4) the SU "Poly-Set" process; (5) a laboratory "silicone fiber encapsulation"; (6) a laboratory urethane coating process involving back-coating with a crosslinking treatment; (7) face-coating with APO tris-(1-aziridinyl)phosphine oxide followed by back-coating with Permafresh 183 crosslinking agent; and (8) crosslinking with epichlorohydrin with the fabric in the flat state, followed by ironing after backcoating with Permafresh 183 crosslinking agent to set the crease. Both electron micrographs of fiber surface replicas and observations with the scanning electron microscope were used in these evaluations.

It is difficult with reflected illumination to obtain good low-magnification photomicrographs of abraded fabric surfaces because of the diffuse scattered reflection set up by the frayed and broken fiber ends. The scanning electron microscope, having 300 times the depth of focus of the low power light microscope, permits a better view of abraded fiber tips simultaneously with that of a considerable length of undamaged fiber on either side of the abraded spot in the yarn. These scanning views establish a better perspective of the relative amounts of abraded fiber in a given area of the fabric, but they fail in illustrating the mechanisms of wear, because they do not have sufficient magnification to reveal the fate of fibril assemblies within the fiber. Transmission electron micrographs extend these observations by permitting examination of structural detail in surface replicas of damaged fibers.

The surface of the untreated cotton fiber is characterized by wrinkles and grooves running at a slight angle to the fiber axis.

In crosslinked cottons these rugosities are still largely evident, although of reduced height and depth. In fibers coated with urethane or silicone, the grooved surfaces are obliterated, and the surface of such coated cotton fibers resembles the nondescript smooth topography of synthetic fibers.

Untreated cotton fibers when flex-abraded dry, as on the Stoll abrader, usually splinter or fracture in a ragged fashion, showing bruised, mangled, and crushed areas along the length of the fiber near the rupture; fiber ends are frequently frayed. When untreated cotton fibers are flexed wet in the laboratory abrader, or in machine washing and tumble drying, the surface wrinkles are smoothed out, the primary wall is often pushed aside or rubbed off entirely, fibrillation occurs, and the hairlike fibrils of the secondary wall cellulose are loosened and lie in tangled masses on the fiber surface.

In crosslinked cotton, the most typical feature of dry flex-abrasion is the development of cracks entirely across the axis of the fiber, the appearance of incipient cracks, faults, and dislocations in the body of the fiber, and the clean-cut break of fiber ends without bruising or fibrillation. Characteristic also of flex-abrasion of crosslinked cotton are "pinch-fractures" along the outer edges of fibers, and the displacement of relatively large hunks of material as they crack out of the body of the fiber.

Wet-abrasion of crosslinked cotton fibers is well illustrated by the surfaces of Permafresh treated, previously coated fibers, after 20 cycles of washing and tumble drying. The fibrillation and shredding typical of untreated cotton fiber under conditions of wet-abrasion is modified. The wet-abrasion of crosslinked fibers takes the form of peeling back of wide tapes or ribbons of cellulose that fracture at sharp angles, to accumulate in sizable fragments adhering to the fiber or depositing on its surface or in surrounding areas.

Accelerotor-abrasion appears to be of a different character. Its features involve chopping of chiplike fragments from the fiber surface and gouging out of chunks randomly, without relation to the underlying fibrillar structure of the fiber.

In crosslinked cotton, this chipping is reminiscent of disintegrating shale rock, with accumulation of flakes stacked upon the fiber surface.

Examination of trouser cuffs, which had been washed and tumble dried through 20 wash cycles, revealed a variety of the above-described effects. Every type of abrasive damage could be seen in every sample examined, but certain characteristics were more pronounced in some than in others. In abraded areas surrounding holes worn in untreated cuffs which had been washed and tumble dried, the fraying of the fiber topography was particularly prominent. Surprisingly, it was even more pronounced on the flat areas of the trouser leg well above the cuff point and away from the crease. As a matter of fact, the replicas made on the trouser crease were disappointingly sparse in damaged fibers; fibers damaged in the abrasion of creases to the failure point had broken out and been lost; and only those fibers initially protected remained for observation and they showed little damage.

In the fabrics that had been face-coated and then back-coated with the Permafresh 183 crosslinking agent, abrasion was apparently delayed; but when abrasion occurred, it was as typical of crosslinked cotton as if the initial face-coating had not been present. Samples of crosslinked cotton tumble dried for 20 cycles without washing showed fractures typical of dry flex-abrasion on the Stoll abrader.

FIFTH SESSION: G. Bloomquist, Swift Spinning Mills, Chairman

IMPROVED PRODUCT QUALITY WITH NEW SRRL BLENDER

[Summary]

by
J. I. Kotter and H. L. Salaun, Jr.
Cotton Mechanical Laboratory
Southern Utilization Research and Development Division

and
C. L. Beaudrot
Greenwood Mills
Greenwood, S. C.

(Presented by H. L. Salaun, Jr.)

The SRRL Bale-Opener-Blender is a machine for opening and blending cotton at textile mills from as many as 20 bales. The machine utilizes a specially formed continuous bale made up of interlaced layers from each of the bales of cottons to be blended

A processing assembly with two counter-rotating toothed cylinders, vertically traverses the end of the composite bale every 2 seconds extracting small tufts from each layer. Tuft weights range between 0.2 and 0.5 g. with production rates up to 1,000 lb. an hour. At higher production rates tuft weights will be slightly higher.

The machine is hydraulically powered with the exception of an electrically driven doffer. The use of hydraulics simplifies machine design and provides precise adjustments of speed and output force in each machine function. Except for periodic manual loading, the machine is fully automatic. It can be operated continuously or on a demand basis, controlled by processing lines.

A comprehensive mill evaluation conducted at Greenwood Mills Durst Plant, Greenwood, S. C., has shown that processing can be accomplished through use of the Bale-Opener-Blender (BOB) with fewer machines in the opening room, while obtaining an improved product. Waste checks at opening, picking, and carding showed that 12 percent more total waste was removed by the standard mill line than the "BOB" line. The lint in the waste, however, was 22-1/2 percent making overall trash removal through carding about equal for the two lines. The uniformity of the finisher drawing sliver was the same for the BOB and standard mill lines.

Neps at the card were about equal for the two lines. Yarn strength showed little, if any change, while imperfections increased slightly. Fabric strength was increased and, even more significant, the fabric grading showed a superior end product. In three separate evaluations, the BOB fabric averaged 25 percent better than that processed by standard mill lines. With proper adjustment of processing machinery to optimize performance on stock delivered by the BOB, the results should be even more promising.

DISCUSSION

Question: Could the blender be used to blend synthetics with cotton?

H. L. Salaun, Jr.: Blending synthetics with cotton has not been tried.

DESIGN OF DURABLE-PRESS SEERSUCKER FABRICS FOR MEN'S SUITS

by
G. J. Kyame, J. T. Lofton, and J. J. Brown
Cotton Mechanical Laboratory
Southern Utilization Research and Development Division

(Presented by J. T. Lofton)

ABSTRACT

The development of an all-cotton, durable-press seersucker suiting material at the Southern Regional Research Laboratory (SRRL) is described. Also presented are data to show the effect of cost-reducing, specification changes on the laboratory performance of the modified fabric.

INTRODUCTION

Seersucker, also known as crinkle cloth, differs from other fabrics in that its structure is one of alternating flat and stripes. It is a wash fabric that is easily woven on any power loom adapted to weaving light or medium-weight fabrics. In weaving seersucker, two warp beams are necessary, one to supply the yarns for the flat, ground stripe, and the other for the crinkle stripe.

Seersucker reached the height of popularity in men's wear during the "roaring twenties" as the ideal summer wear in hot, humid climes. By the early thirties, men's seersucker suits were fast disappearing; probably due in part to the introduction of the "plisses"--seersucker-type fabrics in which the crinkle stripe is produced by chemically induced fabric shrinkage.

Today, a revival appears in the making with the recent public acceptance of striped seersucker sports jackets. This poses the question: Can the seersucker suit stage a comeback with the aid of durable-press properties? The answer is being sought in research now underway at SRRL. This is a report on the results of that research.

Fabric Evaluation

In early experiments at SRRL, textile technologists, using yarns made from medium staple cotton, designed and wove a seersucker fabric which performed promisingly in laboratory tests. By changing only the cotton variety used, a fabric was woven for service testing and additional laboratory evaluation. This gray and white-striped seersucker, 78 x 60 construction, was woven from combed Pima S-2 cotton yarns, 40/2 (15 x 2 tex) in the warp and 60/2 (10 x 2 tex) in the filling. Eight black dyed warp yarns alternated with eight bleached white yarns to form the gray-white striped pattern. These yarns were mercerized at the Laboratory in a beam-to-beam operation using the slack mercerizing-post tensioning technique developed by Murphy and coworkers[1] The filling yarns were white bleached Pima S-2 yarns mercerized by Dixie Yarns, Inc., Chattanooga, Tenn.

A Draper XP loom, equipped for dual-beam operation with two positive beam let-offs, was used to weave the experimental fabric. Crimp in the white stripe was obtained by adjusting the white beam let-off to give an on-loom warp contraction[2] of about 27 percent; corresponding contraction for the tight, black warp was about 7 percent. Two hundred yards of the seersucker were submitted for commercial finishing and service testing.

A sample of the commercially finished fabric was evaluated at the Laboratory for physical properties and the type of wear caused by home laundering. One portion of the sample was pressed flat and cured in conventional manner for the physical properties determinations. The remainder was fabricated into three, short length, cuffed trouser legs, which were pressed, cured, and then tested for wearability by repeated laundering. In the latter test, the cuffs were subjected to 30 wash-wear cycles of home laundering and tumble drying under procedures recommended by the American Association for Textile Chemists and Colorists (AATCC)[3]

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[1] Murphy, A. L., Margavio, M. F., and Welch, C. M. A Method of Preventing Strength Losses during Wash-Wear Finishing of Cotton Fabric. Amer. Dyestuff Rptr. 53: 42-43. 1964.

[2] Kingsbery, E. C., and Roddy, N. P. Method Used for Determining Warp Crimp. Textile Res. J. 29: 521-522. 1959.

Laboratory performance of mill-finished, experimental seersucker fabric are given below:

Fabric weight	5.1 oz./sq. yd.
Thread count (WXF)	82 x 64

Crimp:

Black warp	4.5 percent
White warp	33.3 percent
Filling	8.3 percent

Breaking strength:

Warp	38 lb.
Filling	32 lb.

Elongation at break:

Warp	12 percent
Filling	14 percent

Tearing strength:

Warp	tear reverted to crosswise direction of specimen
Filling	1,300 g.
Resin add-on	7.1 percent

Crease recovery:

W+F	289°
Avg. wash-wear appearance (Monsanto replica stds.)	5.0
Avg. crease appearance after 30 launderings (AATCC Photo stds.)	4.7
Avg. holes per cuff after 30 launderings[1]	2.0
Range (holes/cuff)	1 to 3

Breaking strength, elongation-at-break, and tearing strength are believed adequate for use in men's suiting. Crease recovery angle, though not quite so high as desired, is sufficient to give good smooth-drying and crease retentive properties. This can be seen in figure 1, which shows the best and the worst of the three cuffs that were laundered. Wash-wear appearance ratings were determined by comparing against Monsanto Plastic Replica Standards, and crease appearance ratings against AATCC Photographic Standards. Some difficulty was experienced in rating the cuffs because of the influence of the striped pattern; consequently, the figures shown should be interpreted as a qualitative indication of fabric performance rather than as a quantitative measure. Figure 2 shows a service-tested suit on a mannequin.

Effect of Cost-Reducing Fabric Changes

In an effort to reduce production costs, the investigation was extended to determine what would happen if commercially available mercerized yarns made from lower priced cotton were used instead of the specially mercerized Pima yarns. Accordingly, plied and singles mercerized yarns equivalent in yarn number to the previously used Pima yarns were obtained from a commercial source. These yarns and their physical properties are identified in table 1. Included in the table are data for the Pima yarns (yarns Nos. P-1, P-2, and P-3) used in preparing the previously mentioned, laboratory tested fabric.

Four experimental fabrics were woven from the less costly yarns. These, and a sample of the Pima yarn fabric, were given a conventional durable-press finish with Permafresh 183. As before, samples were pressed flat and cured for physical properties determinations and six trouser cuffs were fabricated. The cuffs were pressed, cured, and subjected to 55 launderings. Table 2 shows how these fabrics performed; fabric A in the table is Pima yarn fabric.

The superior wearability of the fabric woven from plied Pima yarns is apparent, although the breaking and tearing strengths of this fabric were no better than those of the best of the mixed yarn fabrics. In the latter, a changeover from ply to singles yarn is usually accompanied by a drop in the breaking, tearing, and bursting strengths. Fabric wearability also shows progressive deterioration with singles yarn substitution, but the effect is much more pronounced if the changeover is made in the filling yarns.

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[1] Maximum hole diameter 3/32 in.; hardly discernible in view of stripped pattern.

[3] Amer. Assoc. Textile Chemists and Colorists. Appearance of Fabrics in Wash-and-Wear Items after Home Laundering. Tentative Test Method. AATCC Tentative Test Method No. 88 A–1964T, Tech. Manual 41: B–99–B–103. 1965.

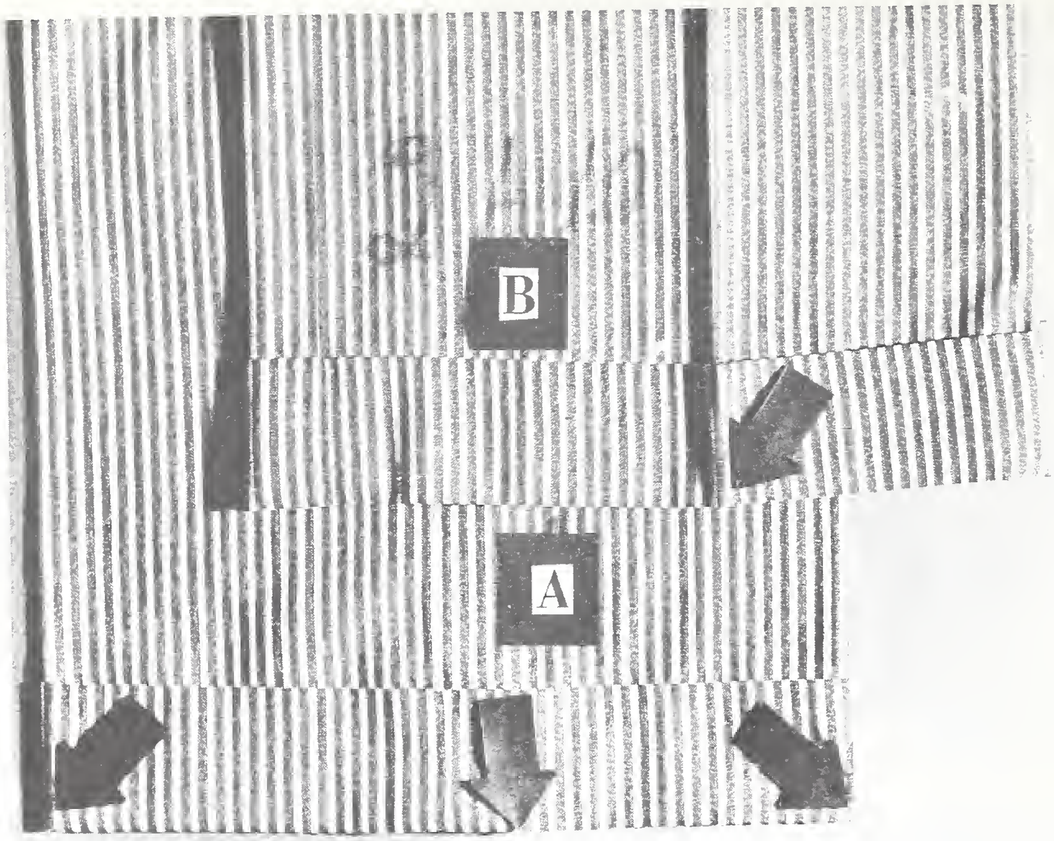

Figure 1.—Best and worst of three cuffs laundered.

Figure 2.—Service-tested suit.

Table 1. --Properties of the mercerized yarns used in the tests

Yarn identification	Nominal yarn number	Cotton Source	Cotton Staple	Cotton Grade	Color	Elongation-at-Break Percent	Break factor oz.
P-1	40/2	(1/)	(1/)	(1/)	Natural	4.21	539
P-2	40/2	(1/)	(1/)	(1/)	Black	5.80	546
P-3	60/2	(1/)	(1/)	(1/)	Natural	4.30	313
P-4	40/2	Calif.	1-1/16"	S. L. Brt.	Black	4.55	445
P-5	60/2	El Paso	1-5/32"	M	Bleached	4.05	480
S-1	20/1	Delta	1-3/32"	S. L. Brt.	Bleached	5.55	284
S-2	20/1	Delta	1-3/32"	S. L. Brt.	Black	5.05	326
S-3	30/1	Delta	1-3/32"	S. L. Brt.	Bleached	4.55	200

1/ Pima S-2 yarns; history not known.

Table 2. --Physical properties and performance of the experimental seersucker fabrics

Sample	Yarns used 1/ Tight warp	Yarns used 1/ Crimp warp	Yarns used 1/ Filling	Fabric construction WXF	Fabric weight	Breaking strength W	Breaking strength F	Elongation-at-break W	Elongation-at-break F	Tearing strength W	Tearing strength F	Bursting strength W F	CRA W+F	Wearability 2/ Average holes per cuff
					oz./sq.yd.	Lb.	Lb.	Pct.	Pct.	G.	G.	Lb.	Degree	
GREIGE FABRIC														
A	P-2	P-1	P-3	78x61	4.64	60	56	12	11	(3/)	4667	--		
B	P-4	S-1	P-5	86x64	5.25	62	60	22	22	3800	3133	99		
C	S-2	S-1	P-5	85x63	5.47	44	61	23	22	2700	2667	83		
D	P-4	S-1	S-3	81x64	5.16	57	32	21	17	(3/)	920	52		
E	S-2	S-1	S-3	81x64	5.25	45	31	23	16	2125	747	45		
FINISHED FABRIC (18 PERCENT PERMAFRESH 183)														
A	P-2	P-1	P-3	82x63	5.11	45	32	12	14	(3/)	1633	--	289	0.5
B	P-4	S-1	P-5	85x61	4.95	51	45	13	17	2650	1816	63	284	1.7
C	S-2	S-1	P-5	86x61	5.29	32	42	13	17	1867	1450	47	298	2.2
D	P-4	S-1	S-3	81x61	4.95	49	21	13	12	(3/)	407	32	279	7.8
E	S-2	S-1	S-3	82x61	5.06	33	18	12	13	(3/)	327	29	298	4.5

1/ See table 1 for yarn identification; "P" indicates yarn, "S" indicates singles yarn.
2/ Data after 55 launderings; no test conducted on the greige fabrics.
3/ Tear reverted to crosswise direction of specimen.

Test Marketing

A fabric made up by a commercial source substantially to the specifications of fabric A, table 2, was tailored into men's suits and test marketed in six major cities in the summer of 1966. The favorable results obtained have led several converters to express a strong desire to offer all-cotton durable-press seersucker suitings. This market program was sponsored by the National Cotton Council of America as part of its research and promotion activity on cotton summer suits.

Summary and Comment

The design and evaluation of a durable-press, all-cotton seersucker fabric has been described. It was shown that substitution of lower cost yarns for plied Pima yarns reduced the fabric's durability in the washing machine.

While a limited marketing study was apparently successful, research continues in an effort to improve performances and lower cost of seersucker fabrics.

ACKNOWLEDGMENTS

The authors are indebted to many of their coworkers for the preparation, finishing, and testing of the experimental fabrics. They wish to express their appreciation to Messrs. M. L. Bocage, N. P. Roddy, and J. J. Landry, in the Cotton Mechanical Laboratory, and to the many cooperating members of the Cotton Finishes Laboratory's Weather Resistance Investigations and Cotton Physical Properties Laboratory's Textile Testing Investigations. Particular thanks go to the National Cotton Council of America and its affiliate, the Cotton Producers' Institute, for partly financing the study and for invaluable advice on industry and marketing problems.

THE EFFECT OF FABRIC STRUCTURE ON DURABLE PRESS PERFORMANCE

by
G. J. Kyame, J. T. Lofton, and G. F. Ruppenicker, Jr.
Cotton Mechanical Laboratory
Southern Utilization Research and Development Division

(Presented by G. J. Kyame)

Introduction

Research was conducted at the Southern Regional Research Laboratory to obtain basic information on the effect of certain yarn and fabric parameters on the durable press properties of crosslinked, summer weight, cotton suiting fabrics made from plied yarns. For the study, 140 experimental fabric structures were prepared. The performance of these fabrics in physical testing and in the washing machine are presented.

Materials and Methods

Yarns for the experimental fabrics were spun from a blend of three bales of 1-3/8 in., 3.8 micronaire, Pima-S-2 cotton of normal preparation. Singles yarns, 60/1 (10 tex) and 40/1 (15 tex), were each spun to 3.00 T.M. and 3.75 T.M. The four yarns resulting were then plied to make the following yarns: (a) 60/2 (10 x 2 tex), 3.00 T.M.; (b) 60/2 (10 x 2 tex), 3.75 T.M.; (c) 40/2 (15 x 2 tex), 3.00 T.M.; and, (d) 40/2 (15 x 2 tex), 3.75 T.M. These yarns and the fabrics woven therefrom are shown in figure 1. Twelve separate rolls of fabric were woven, each roll containing all structure made from the same yarn and same thread count. The fabrics were scoured and bleached in a dye beck and dried on a tenter frame under minimum warpwise tension and with a 2 in. fillingwise shrinkage allowance. The bleached fabrics were padded to 65 percent wet pickup in a solution of Permafresh 183 formulated as follows:

Permafresh 183	18	percent
Mykon S. F.	4	percent
Catalyst X-4	3.2	percent
Water to make	100	percent

The padded fabrics were air-dried on the tenter frame at minimum tension settings.

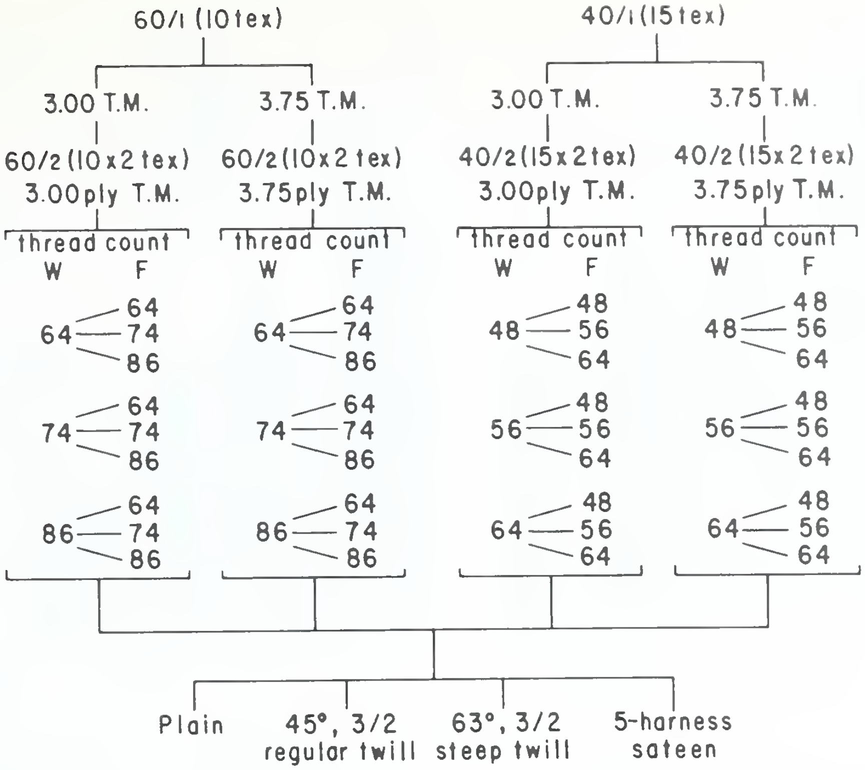

Figure 1.—Fabric organization chart.

Test Procedures

For the physical properties determinations, 1-yard samples of each of the treated fabric structures were pressed flat in a steam-heated, hot-head press for 6 seconds at 90 pounds' pressure, and cured in a circulating oven for 8 minutes at 320° F. The pressed and cured fabrics were given a single washing to remove unreacted resin and catalyst, then tumble-dried. American Society for Testing and Materials (ASTM) test procedures[1] were followed in making physical properties determinations on these fabrics.

Four, short-length (12 in.), cuffed, trouser legs were made from each of the experimental fabrics for use in determining the performance of the fabrics in the washing machine. The cuffs were pressed and cured as described above, and then subjected to 80 cycles of washing and tumble-drying. The wash cycle used was one recommended for "cotton and colored goods" (designated "cycle B" on the machine). The cuffs were examined for holes after each five cycles of laundering. The rupture of a single yarn, warp or filling, in either the cuff or in the creases was classed as a hole and so counted. Holes developing in the hemmed, upper edge of the cuffs were not counted. At the end of the test, the cuffs were rated for wash-wear appearance and sharpness of creases. Rating procedures followed substantially those recommended by the American Association of Textile Chemists and Colorists (AATCC)[2]. The test standards used were: Monsanto Plastic Replicas for rating the wash-wear appearance, and AATCC Photographic Standards for rating crease appearance.

Results

Although this experiment is a relatively small one, considering the magnitude of the subject under study, the quantity of data obtained is considerable. The bar chart (figure 2) gives an overall picture of the performance of the experimental structures in

[1] American Association of Textile Chemists and Colorists. AATCC Tentative Methods 88A–1964T and 88C–1964T, Tech. Manual 41: B99–B103, B104–B105. 1965.

[2] American Society for Testing and Materials, Philidelphia, Pa. ASTM Designation: (a) D 1295–53T: (b) D 1682–59T; (c) D 1424–59; (d) D 1175–55T. 1966.

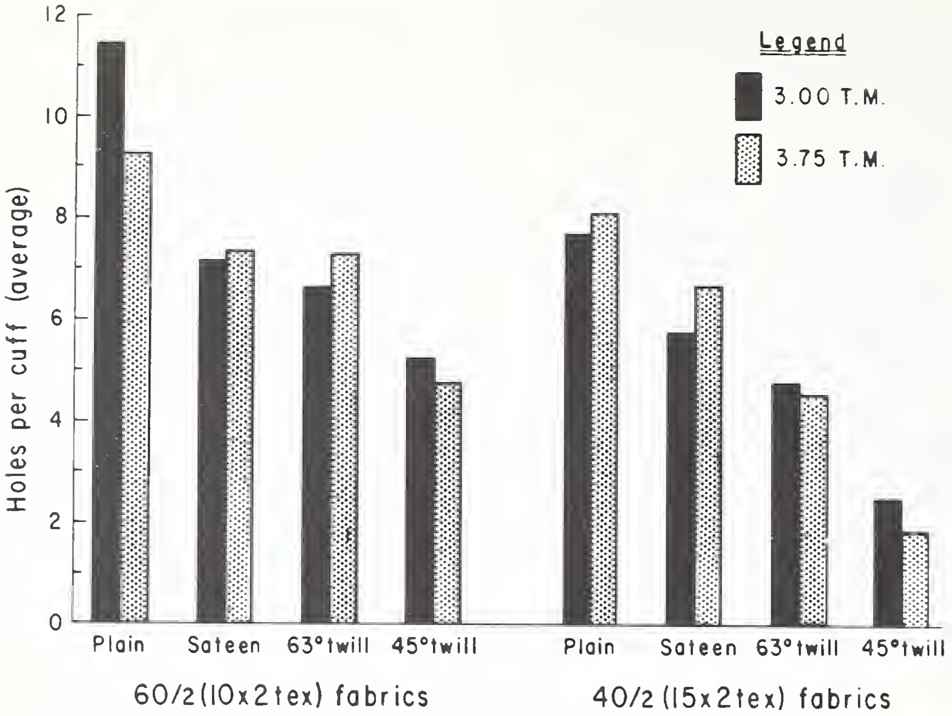

Figure 2.—Performance of the fabrics in the washing machine.

the washing machine. The chart shows the average number of holes per cuff developed after 80 launderings in each group of fabrics made from the same yarn and same weave type. Greatest wear occurs in the plain weave fabrics, and least wear in the 45° regular twills. This is true for the 60/2 (10 x 2 tex) fabrics, as well as for the 40/2 (15 x 2 tex) fabrics. The 63° steep twill fabrics were slightly better than the sateens, the differences being more pronounced in the 40/2 (15 x 2 tex) fabrics. Yarn twist shows no significant effect on fabric wear, leastwise not within the limited range studied. Although the mean crease angles for each fabric group differ by as much as 21°, there are sufficient inconsistencies between the corresponding wear values to preclude explanation of the observed performance on the basis of crease angle alone. On the whole, the 40/2 (15 x 2 tex) fabrics performed better than the corresponding 60/2 (10 x 2 tex) fabrics did.

The effect of yarn thread count is shown in table 1. An increasing thread count, whether warp or filling, has an adverse affect on fabric wear. The discrepancy noted in the middle warp thread count for the 40/2 (15 x 2 tex) fabric group is believed due to the effect of the comparatively low resin add-on applied to this group.

Overall averages of some of the physical properties measured are given in table 2. Separate averages for the 60/2 (10 x 2 tex) fabrics and for the 40/2 (15 x 2 tex) fabrics are shown. Included in the tables are simple correlation coefficients which indicate the correlation between the property listed and the number of holes worn into the cuffs after 80 launderings. These correlations were computed from the combined data for all 144 fabrics. In most instances, the data reveal little difference between the mean properties of the two sets of fabrics. Although the 40/2 (15 x 2 tex) fabrics were 15 percent heavier than 60/2 (10 x 2 tex) fabrics, the extremely low correlation coefficient calculated for this property indicates no effect of fabric weight on fabric wear. The highest correlation coefficient is that for filling tearing strength. On the whole, the 40/2 (15 x 2 tex) fabrics, in developing 27 percent fewer holes, outperformed the 60/2 (10 x 2 tex) fabrics in the repeated laundering test.

The fabric constructions for this experiment were planned to yield pairs of fabrics matched in weight, but differing primarily in the size of the yarns from which they were made. Shrinkage during finishing, however, resulted in only four groups of fabrics sufficiently similar in weight and thread count to

Table 1.--Fabric wear as related to fabric thread count

Yarn No.	Wear after 80 launderings					
	Warp count			Filling count		
	Avg. ends/inch	Avg.[1] holes/cuff	Avg. pct. resin	Avg. picks/in.	Avg.[1] holes/cuff	Avg. pct. resin
	71	5.1	5.1	67	6.8	5.1
60/2	82	8.4	4.9	77	7.3	5.1
(10 x 2 tex)	96	8.7	5.2	89	8.1	5.0
	54	5.1	6.3	50	4.6	5.9
40/2	63	3.6	5.1	58	5.2	5.9
(15 x 2 tex)	70	7.0	6.2	65	6.0	5.8

[1] Each figure is the average of 96 observations made on four cuffs of each of 24 fabric structures representing four weaves, two twist multipliers, and three thread counts.

Table 2.--Fabric properties and their relation to fabric wear in the washing machine

Property[1]	Overall averages		Correlation coefficient[2]
	60/2 (10x2 tex)	40/2 (15x2 tex)	
Weight . . . oz./sq. yd	3.9	4.5	0.09
Breaking strength:			
Warp . . . . lb.	40	42	.36
Filling . . . lb.	32	33	.36
Elongation-at-break:			
Warp . . . . percent	8.4	8.8	.31
Filling . . . percent	17.4	20.1	.12
Tearing strength:			
Warp . . . . g.	1,853	2,741	-.48
Filling . . . g.	1,579	2,599	-.65
Holes per cuff after 80 launderings	7.3	5.3	--
Resin add-on:			
Before laundering . . . . percent	5.1	5.9	-.17
After laundering . . . . percent	4.4	5.3	-.18
Crease recovery angle (W+F):			
Conditioned . . . . degree	288	284	.30
Wet . . . . degree	252	250	.01
Wash-wear appearance:			
Monsanto stds. rating after laundering	4.1	4.2	-.29
Crease appearance:			
AATCC Photo. stds. rating after laundering	3.9	3.8	.53
Holes per cuff after 80 launderings	7.3	5.3	--

[1] Finished fabric.

[2] The relationship between the property named and the number of holes worn into the test cuffs by launderings.

permit making the desired comparisons; the data for these are given in figure 3, which compares the wear of the fabrics by weave type. Compared within each of the four groups are eight fabrics made from 60/2 (10 x 2 tex) yarns and eight from 40/2 (15 x 2 tex) yarns. The mean weights and mean thread counts listed are the averages of the data for each subgroup of eight fabrics. Two facts are apparent: (1) Weave type plays a prominent part in determining fabric wear, and (2) fabrics made from the heavier 40/2

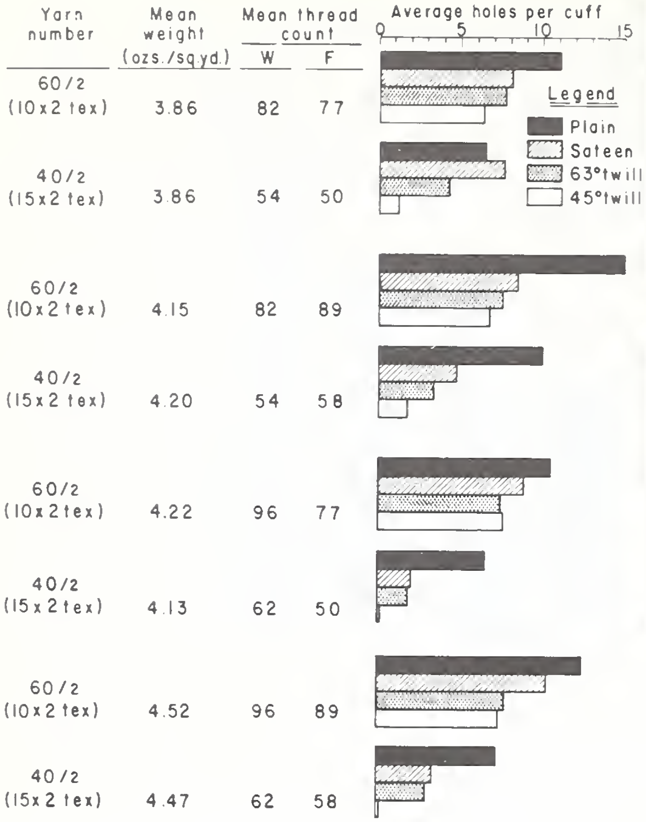

Figure 3.--Performance of fabrics of comparable weight in the washing machine.

(15 x 2 tex) yarns outwear those of comparable weight made from the lighter 60/2 (10 x 2 tex) yarns. In general, the plain-woven fabrics exhibited the greatest wear and were followed in order by the sateens, the 63° steep twills, and with the least wear, the 45° regular twills.

Discussion and Conclusions

The wear performance of a fabric is closely associated with the freedom of its yarns to move about within its structure and with the degree of exposure of these yarns to the wear causing forces encountered in use. The good wearing qualities of the 45° twills are believed due to a combination of freedom or yarn movement within their structures and a near balance in exposure of the warp and filling yarns to the wearing forces. The plain weave, on the other hand, severely restricts movement of the yarns within the fabric's structure. Thus, this lack of yarn mobility overrides the balanced exposure of the yarns to wear. This is more readily seen in figure 4, which shows the patterns of the weaves studies. In the sateens, extreme exposure of the warp yarns overrides the high yarn mobility inherent in this type of structure and accounts for its relatively low resistance to wear.

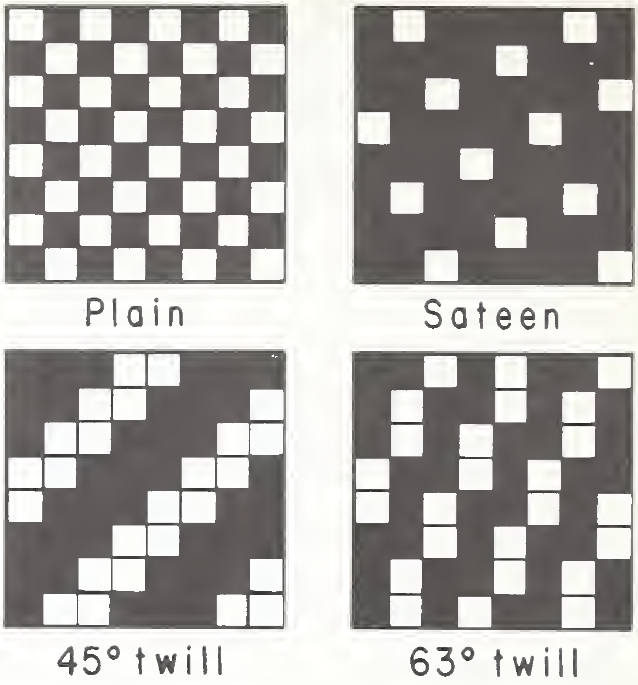
Plain
Sateen
45° twill
63° twill

Figure 4.--Fabric weave patterns.

Ordinarily, one would expect the 63° steep twills to perform as well as the 45° regular twills since both are 3/2 twills. A comparison of the weave patterns, however, shows that the filling yarn exposure in the steep twill is subdued by the plain-weavelike interlacing, thus leading to the greater wear observed.

Research in Progress

A similar study in which equivalent singles yarns were substituted for the ply yarns is now nearing completion. Test trouser cuffs for this study had to undergo 115 launderings before fabric breakdown occurred in all experimental structures but one. Statistical computations on the data obtained are in process. The investigation is being extended into the field of 7-9 oz./sq. yd. suiting fabrics made from medium staple cottons.

ACKNOWLEDGMENTS

The authors wish to express their sincere appreciation to the many coworkers in the Cotton Mechanical Laboratory for valuable

services rendered in preparing the experimental fabrics. They are indebted also to the many cooperating members of the Cotton Finishes Laboratory's Weather Resistance Investigations and of the Cotton Physical Properties Laboratory's Textile Testing Investigations for their contributions in finishing and testing the fabrics. Special thanks go to Herschel W. Little, Statistician in CML, for planning the test and analyzing the data, and to the Cotton Producers' Institute for their financial support of the study.

DISCUSSION

Question: In examining the cuffs after laundering, was it noted in the sateen fabrics which of the yarns, warp, or filling failed first?

G. J. Kyame: No attempt was made to classify fabric failures in such detail because the experiment was directed toward determining the interactions between the various parameters preparatory to designing more detailed experiments which would take into account the observed interactions.

Question: Research in duPont's laboratories has indicated that fabric stiffness plays an important part in determining the fabric's resistance to wear. Have we looked at this property?

G. J. Kyame: Stiffness was considered in our study and stiffness measurements were made on a selected group of the experimental fabrics. The resulting data showed no consistant patterns of behavior so they were not included in the report.

Question: The military uses a lot of sateen fabric and they have found that by putting the filling face of the fabric to the outside of the garment the wear life of the garment is increased. Would SRRL have gotten different results if cuffs were tested with the sateen filling face on the outside?

G. J. Kyame: The fabrics in the study were lighter in weight than those used by the military. What the results would have been if the sateen cuffs were tested with the filling face out is unknown. However, the studies are being extended to heavier fabrics. Perhaps this factor can be investigated.

RELATIONSHIPS AMONG FIBER PROPERTIES, YARN PROPERTIES AND END BREAKAGE

[Summary]

by
G. L. Louis, L. A. Fiori, and L. A. Leitz
Cotton Mechanical Laboratory
Southern Utilization Research and Development Division

(Presented by L. A. Fiori)

Seventy-nine medium staple length cottons with a wide range of fiber properties were investigated to determine the (a) ranking of fiber properties in their order of importance relative to yarn properties and end breakage; (b) ranking of yarn properties in their order of importance relative to end breakage; and (c) means to predict yarn properties and end breakage from fiber and/or yarn properties.

The cottons were processed using a similar processing organization. Carding rate was 20 pounds per hour except for the low Mirconaire reading cottons which were carded at a lower rate. The cottons were spun into 18/1 (33 tex), 22/1 (27 tex), and 36/1 (16 tex) yarns at twists for maximum strength, and also with a range of twists. Yarn properties and end breakage rates were obtained. End breakage rates were measured by the SRRL 720 spindle hour and Accelerated spinning tests.

It was found that the 50 percent span length is a better basis than the 2.5 percent span length for use in explaining the effects of fiber length on yarn properties and end breakage. Fiber tenacity, measured at 1/8" gage length, contributes most toward yarn strength while fiber length contributes most toward end breakage in spinning. Generally, fiber length distribution expressed by the center of gravity of length technique yielded the highest relationship when correlated with yarn properties and end breakage, as compared with other fiber length distribution criteria. Single strand strength ranked first, yarn uniformity second, and yarn elongation third in importance in affecting end breakage. On an average about 81 percent of the variation in yarn strength may be explained by or attributed to the fiber properties evaluated. About 72 percent (Accelerated spinning test) and 38 percent (720 spindle hour test) of the variation in end breakage may be attributed to fiber properties directly. Yarn properties contributed about 81 percent (Accelerated spinning test) and 48 percent (720 spindle hour test) to the variation affecting end breakage in spinning. Yarn strength and elongation increased linearly with yarn number when expressed in tex, making it possible to predict these properties when two values of yarn strength or elongation spun from the same cotton are known.

DISCUSSION

Question: Is the paper available?

L. A. Fiori: The paper is not yet available. However, it should be in proper form for publication in about 2 months. If you are interested at that time, we will be glad to send you a review copy for your comments.

THE PRACTICAL IMPORTANCE OF FIBER HOOKS TO PROCESSING PERFORMANCE

by
L. A. Fiori, J. Simpson, and L. B. DeLuca
Cotton Mechanical Laboratory
Southern Utilization Research and Development Division

(Presented by J. Simpson)

ABSTRACT

The following basic principles can be used to reduce fiber hooks and improve processing performance:

Carded Yarns:--The "majority" hooks should always trail into spinning and be drafted in the trailing direction in as many of the prespinning processes as possible. When more than two drawing operations are used, hooks may be reduced substantially so that their directional effect at spinning on yarn properties and end breakage may be negligible.

Combed Yarns:--The "majority" hooks should always lead into the combing process and be drafted in the trailing direction in as many of the precombing processes as possible. The combing and postcombing operations reduce hooks to such a low level that their direction in spinning is unimportant and their effect on yarn properties and end breakage is negligible.

Carding Process:--For cottons of low or moderate napping potential, high production carding should result in a reduction in the "majority" hooks and resultant improvement in drawing sliver uniformity, reduction in ends down in spinning, and reduced comber noil percentage.

General:--The major factors of fiber hook formation are mechanical actions during processing, mainly during carding, and total drafts and draft directions. Fiber properties do not control fiber hooks.

Introduction

Fiber hooks have been known to exist in processed cotton for a long time (7, 8, 9, 10, 11). Their effect on processing performance and product quality has not been of practical importance heretofore mainly because the textile industry used comparatively low processing speeds and multiple drawing and roving processes prior to spinning. However, recent trends toward high production carding, high drafts, decreased doublings, increased sliver weights, and automated processing systems have emphasized the need for scientific information on the influence of fiber hooks on processing performance.

Fiber hooks have been a confusing area of research due mainly to the difficulty of interpreting findings and applying the results under practical conditions. This paper discusses factors affecting fiber hook formation, techniques for reducing them, and improving processing performance. The paper also presents practical suggestions to achieve these objectives through use of proper drafting directions and card production rates.

Fiber Hook Terminology

Fiber hook terminology (8, 16) can best be understood by designating the trailing hooks of card web or sliver as the "majority" hooks and the leading ones as "minority" hooks. The majority hooks in a card web or sliver are always used as a reference point for tracking their position in other processes.

Fiber Hook Formation

Although the raw cotton, picker lap, etc., have highly disorientated fibers, fiber hooks are formed preferentially during carding with about 49 percent majority, 20 percent minority, and the remainder unclassified. Insofar as can be determined, most hooks are formed during the doffing operation between the cylinder and doffer by a combination of surface speeds, air currents, and angle of the teeth. When the cylinder begins to "load," hook formation increases due to changes in air currents with the fibers being closer to the surface of the cylinder clothing (2, 8, 19, 20, 24).

Fiber Hook Measurement

Fiber hooks were first measured by tracer fiber techniques (6, 8). Recently, however, a fairly rapid method, referred to as the modified Lindsley method, was developed (5, 14). Briefly, this involves clamping a fiber bundle between fixed jaws, combing and straightening the fibers, and weighing the residual portions. A number of indices (1, 3, 4, 5, 14, 22) are offered describing degree of fiber orientation and hook formation, but currently the "cutting ratio" (13) is commonly used.

Processing Variables Affecting Fiber Hooks

Sliver Weight, Doubling, Draft.--Fiber hook removal decreases at first drawing as card sliver weight increases, independent of draft (15). Increasing the draft at any process generally will reduce fiber hooks. Doublings, as such, have no effect on fiber hook removal.

Carding Variables.--Generally, increases in card production result in decreases in total hooks--with the majority hooks decreasing and the minority hooks increasing (19). The rate of hook change depends on the type of cotton processed.

Figure 1 shows the hook formation pattern of the majority and minority hooks for cottons differing in staple length as a result of different card production rates. The rate of increase in minority and decrease in majority hooks is dependent on type of cotton processed.

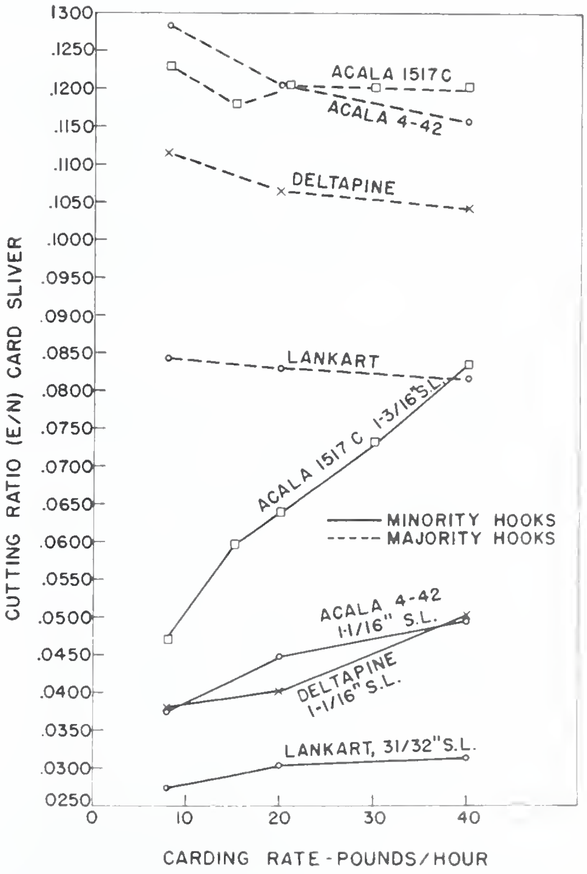

Figure 1.--Effect of card production rate on hook formation for cottons differing in staple length.

The rate of increase of minority hooks with increasing carding rate becomes progressively greater as staple length increases. On the other hand, rate of decrease of the majority hooks with carding rate is about the same for all staple lengths studied. Generally, with short or medium length cottons of low or moderate nepping potential, high carding rates tend to reduce the majority hooks without significantly increasing the minority hooks. Therefore, high card production rates can lead to an improvement in drawing sliver uniformity and consequent reduction in end breakage.

When high card production rates are combined with the proper sliver drafting direction for maximum hook removal, a significant improvement in second drawing sliver uniformity and reduction in end breakage results (19).

Increases in card cylinder speed produce increases in majority hooks, indicating that when the ratio of cylinder to doffer speed increases at any given production rate, the majority hooks increase.

A direct relationship was found between increases in minority hooks and yarn imperfections (20). If the formation of minority hooks could be reduced through changes in carding mechanisms even greater carding production rates might be achieved with an improvement in processing performance and yarn quality.

Drafting Directions

Carding. --The direction in which sliver is drafted at all processes has a significant effect on end breakage and a lesser effect on yarn properties. For minimum end breakage and maximum yarn strength, the majority hooks should trail into the spinning process (12, 13, 16, 20). This requirement becomes more critical as the number of prespinning processes are reduced and yarn number becomes finer (17). Under such conditions, the opportunity for fiber hook removal is limited and the majority hooks could sometimes lead into spinning. If processing sequences are such that the majority hooks lead into spinning, they should be reduced to a minimum by using higher prespinning drafts or an additional drawing process so that end breakage will not be affected adversely.

The amount of fiber hooks entering spinning, whether majority or minority, has a more critical effect on end breakage as spindle speed increases.

Maximum majority hook reduction and minimum end breakage can be obtained by drafting the majority hooks in the trailing direction at first and second drawing, leading into roving, and trailing into spinning independent of cotton qualities used. Figure 2 shows the effect of hooked ends on end breakage when two drafting sequences are used. The control represents the normal drafting direction where the majority hooks are drafted in the leading direction into first drawing, trailing direction into second drawing, leading into roving, and trailing into spinning; the experimental represents the SRRL recommended drafting directions where the majority hooks trail into first and second drawing, lead into roving, and trail into spinning. Increases of about 20 percent in spindle speed and production were obtained with a small increase in yarn strength at constant end breakage when the SRRL drafting direction was used (21).

Drafting removes the minority hooks more efficiently than the majority (16), probably because the majority hooks were somewhat larger (8). On an average, drafting removes between 50-70 percent of the total hooks from card sliver through second drawing and between 85 to 95 percent of the total hooks from card sliver through combed finisher drawing (9, 14, 18, 20).

Combing. --To obtain minimum noil removal during combing the majority hooks should always lead into the comber (10, 18, 23). Additional reduction in noils can also be obtained by reducing the majority hooks leading into combing (10, 20).

Figure 3 shows the effect that the majority hooks have on noil percentage with varying carding production rates and drafting directions. Lots 1C-8, 1C-15, 1C-20, and 1C-40 represent the conventional drafting direction at a carding rate of 8, 15, and 20 pounds per hour for two cottons. Lots 2C-8, 2C-15, and 2C-20 represent the SRRL recommended drafting directions, requiring manual sliver reversals at the drawing and sliver lapper processes, and at the same carding rates. Lot 3C introduces an additional drawing operation and the SRRL draft-

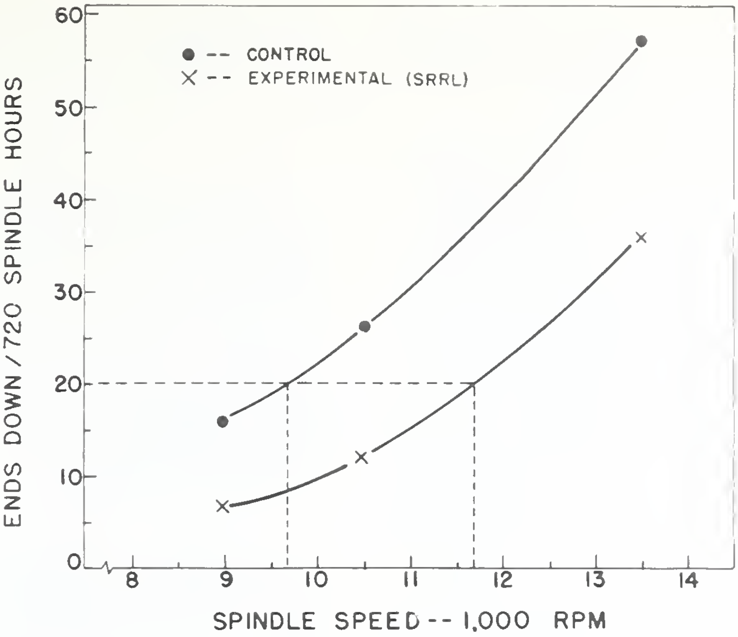

Figure 2.—Effect of hooked ends on end breakage for a range of spindle speeds.

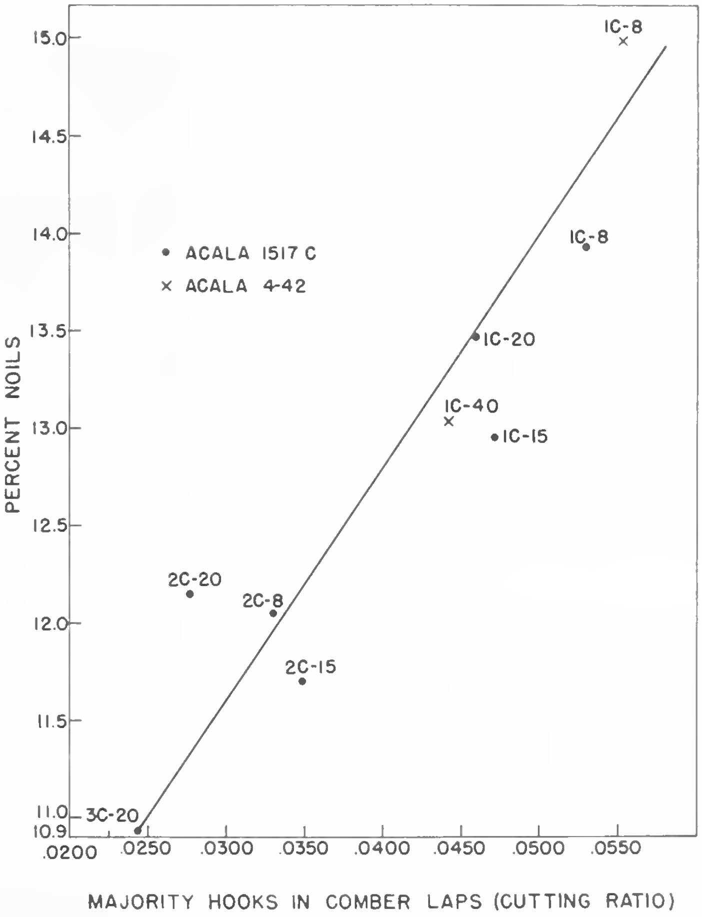

Figure 3.—Effect of the majority hooks on noil percentage at varing carding rates and drafting directions.

ting direction (majority hooks trailing at first drawing, leading at second drawing, trailing at sliver lapper, leading at combing), requiring one reversal at first drawing.

In this connection, the manual reversal of sliver for Lot 3C can be eliminated completely by substituting a Unit Card system followed by a Railway Head which would replace first drawing. The data show (20) that noil percentage can be reduced substantially by reducing the majority hooks leading into combing. This can be done through appropriate combinations of drafting directions represented by Lots 1C, 2C, and 3C, independent of cotton, and by increasing card production rates. For example, over 2 percent noil percentage reduction results at a carding rate of 20 pounds per hour with Lot 3C drafting directions as compared with Lot 1C. Also, noil percentage can be reduced approximately 2 percent by increasing the carding rate from 8 to 40 lb./hr. with the Acala 4-42 cotton.

Reduction of majority hooks to a low level prior to combing also gives better separation of long and short fibers and more uniform sliver, resulting in a reduction in end breakage, particularly at high spinning speeds (20).

Fiber Properties Affecting Fiber Hooks

Fiber hooks are not closely correlated with any of the important fiber properties. The minority hooks were found to be fairly closely and inversely correlated with Micronaire reading. This relationship may be due mostly to the effect of cylinder "loading" during carding that tends to increase the minority hooks, which may explain why a direct relationship exists between minority hooks and neps and yarn imperfections. Generally, the longer the cotton the greater the amount of hooks.

Reduction in fiber hooks from card through second drawing appears to be directly correlated with fiber length and inversely correlated with Micronaire reading. With medium staple cottons of matched fiber properties where bundle elongation was the only fiber variable (25), fiber hooks were reduced during processing more with low than high elongation cottons.

Generally, fiber properties do not control significantly either the formation or elimination of fiber hooks. The major controlling mechanisms are mechanical actions during processing, mainly during carding, and total drafts and draft directions.

Practical Applications

Reducing fiber hooks through the use of proper drafting directions has promising possibilities for decreasing end breakage. However, it is realized that the manual reversing of slivers at the drawing processes required to obtain maximum benefit is impractical. Therefore, available processing sequences were studied with a view to suggesting practical process combinations resulting in maximum hook removal and minimum end breakage.

Tables 1 and 2 list available processing organizations, drafting directions for these organizations, and combinations of drafting directions required for most efficient hook removal for carded and combed yarn.

Carded Yarns

No. 1 Conventional drafting directions wherein the majority fiber hooks trail into spinning as they always should.

No. 2 SRRL recommended drafting directions that result in maximum majority hook reduction, improvements in sliver uniformity, minimum end breakage and maximum yarn strength. The process requires reversing the creels at first and second drawing.

No. 3 Unit card system that does not require manually reversing the creels but only when two drawing operations after the railway head are used, as in (3b). This arrangement results in the maximum reduction of majority hooks since they are drafted twice in the trailing direction.

When only one drawing operation is used after the railway head (3a), the fiber hooks lead into spinning, which is not desirable and could lead to misinterpretation of the performance of the unit card system partly because of the adverse effects of the majority hooks leading into spinning.

Table 1. --Drafting directions, carded yarn

Lot No.	Process		Card	Railway head	First drawing	Second drawing	Third drawing	Roving	Spinning
(1)	Conventional		(—— (1/)		——)	(——		——)	(——
(2)	SRRL		(—— (2/)		(—— (2/)	(——		——)	(——
(3)	Unit Cards	(a)	(——	(——	——)			(——	——)
		(b)	(——	(——	——)	(——		——)	(——
		(c)	(——	(——				——)	(——
		(d)	(—— (3/)	(——	——)				(——
(4)	3 Drawings	(a)	(——		——)	(——	——)	(——	——)
		(b)	(—— (2/)		(——	——)	(——	——)	(——

			Card	Railway head	Pre-blender	Blender	Finisher drawing	Third drawing	Roving	Spin-ning
(5)	Drawing Blender	(a)	(——		——)	——)	(——		——)	(——
		(b)	(——		——)	——)			(——	——)
		(c)	(—— (2/)		(——	(——			——)	(——
		(d)	(—— (4/)	(——	——)	——)			(——	——)

1/ Direction of majority fiber hooks.

2/ Sliver is reversed in cans after indicated process.

3/ Typical drafting direction for automated direct spinning systems.

4/ Unit Cards.

Table 2. --Drafting directions, combed yarn

Lot No.	Process		Card	Railway head	First drawing	Second drawing	Sliver lapper	Comber
(1)	Conventional		(—— (1/)		——)		(——	——)
(2)	SRRL		(—— (2/)		(—— (2/)		(——	——)
(3)	Unit Cards		(——	(——	——)		(——	——)

			Card	Pre-blender	Blender	Finisher drawing	Sliver lapper	Comber
(4)	Drawing Blender	(a)	(——	——)	——)		(——	——)
		(b)	(—— (2/)	(——	(——	——)	(——	——)

1/ Direction of majority fiber hooks.

2/ Sliver is reversed in cans after indicated process.

In (3c) the majority hooks trail into spinning and the majority hooks level is high; but the system might be suitable for yarns requiring only one drawing process. This arrangement has a minimum of doublings and might need some type of automatic "levelling" device at the railway head to control and reduce long-term yarn number variation.

No. (3d), representing drafting directions of a typical automatic direct spinning system, results in the majority hooks trailing into spinning. Introduction of a roving process (not shown) results in the majority hooks leading into spinning, which is not desirable from a hook direction standpoint but desirable for hook reduction.

No. 4 Three drawing system (4a) wherein the majority fiber hooks lead into spinning. However, the additional third drawing operation reduces the hooks to a level where their detrimental effect on end breakage may not be serious. There is also the advantage of additional doublings and resultant reduction in long-term variation in yarn number.

A real advantage with three drawings would be obtained if the card sliver were reversed (4b). Then the majority hooks trail into spinning, and also are at a low level since they are drafted in the trailing direction at two drawing processes.

No. 5 Drawing system for blending, which is a preblender (first) and blender (second) drawing process hooked together in one machine.

In (5a) a preblender, blender and finisher drawing are used resulting in three drawing operations plus the additional advantage of having the majority hooks trailing into spinning. The disadvantage is that the majority hooks are trailing at only one of the three drawing processes.

With some blends and coarse yarn numbers, the temptation might be to eliminate the finisher drawing, as illustrated in (5b). However, this is one of the worst drafting directions because the majority hooks would lead at the preblender, blender, and into spinning.

If, on the other hand, the card slivers were manually reversed, as in (5c), the majority hooks would trail at both the preblender and blender, lead at roving and trail into spinning. This arrangement could then probably be employed for any yarn number.

Where a unit card system precedes the drawing blender (5d), the majority hooks lead twice in the preblender and blender and lead into spinning which is undesirable. However, this involves three drawing processes which might reduce the hooks to such a low level that end breakage may not adversely be affected.

When developing improved processing organizations for carded yarns, the combination of number of processes and drafting directions should be selected so that the majority hooks trail into the spinning process and as many prespinning processes as possible.

Combed Yarns

No. 1 Conventional method of processing combed products, where the majority fiber hooks lead into the comber as they always should.

No. 2 SRRL recommended drafting direction results in substantial reductions in noil percentage, because the majority hooks are reduced more than in the conventional processing method, by being drafted in the trailing direction at all precombing processes. Increasing card production will result in further reduction in noil percentage because of additional reductions in the majority hooks.

No. 3 Unit card system where the best combination of drafting direction and noil percentage removal can be obtained without need of manually reversing the sliver. If high production rates are also used with this combination, substantial savings in noil percentage removal should result over the conventional method No. 1. The main advantage of this system is that the majority hooks are drafted in the trailing direction at both the railway head and sliver lapper without manually reversing of slivers.

No. 4 Drawing blender system which is a preblender (first) and blender (second) drawing process hooked together in one machine followed sometimes by a finisher drawing operation.

In (4a) the majority hooks would lead into the comber which is the correct direction; however, the majority hooks would lead at both the preblender and blender which would not produce maximum hook reduction and minimum noil percentage.

Substantial savings in noil percentage would probably be obtained by using (4b) involving a reversal of the card sliver and an additional finisher drawing process. The majority hooks would trail at the preblender, blender, and sliver lapper and lead into the comber.

Whether a second or third drawing operation is used after combing is not important insofar as fiber hooks are concerned, because the combing and postcomber operations reduce them to such a low level that their effect on yarn properties and end breakage is negligible. Therefore, it is immaterial whether the majority of minority hooks lead or trail into spinning. The number of drawing operations after combing would have an important bearing on doublings and consequently long-term variation effects, especially on yarn number, and could be used to justify elimination of double creel spinning.

From a cost standpoint reduction in noil percentage is always desirable. However, this advantage must be weighed against possible disadvantages such as increased yarn imperfections when carding rate is increased, adverse aesthetic fabric properties, etc., so each manufacturer must decide whether the reduction in noil percentage is economically worthwhile.

When developing improved processing organizations for combed yarns, the majority hooks should always lead into combing, but should trail at as many precomber processes as possible to obtain maximum reduction of the majority hooks.

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ATTENDANCE LIST

Aguillard, Wilbur, Cotton Fiber Testing Laboratory, Louisiana State Univ., 4125 Gourrier St., Baton Rouge, La. 70808.

Allen, Dwight F., United Merchants & Mfgs., Inc., P. O. Box 2148, Greenville, S. C. 29602.

Allen, T. C., Pennsalt Chemicals Corp., King of Prussia, Pa. 19406.

Anello, Louis G., Industrial Chemical Division, Allied Chemical Corp., Morristown, N. J. 07960.

Ansin, Leonard M., Textiles, Inc., 168 Stevens St., Fall River, Mass. 02722.

Anzuoni, Albert R., Market Research Dept., A. E. Staley Manufacturing Co., P. O. Box 151, Decatur, Ill. 62525.

Armstrong, Carl A., Waldrich Bleachery, Clifton, N. J. 730 Walker St., New Orleans, La. 70124.

Baer, Emil, Apex Chem. Co., Inc., 200 South First St., Elizabeth, N. J. 07206.

Barber, Richard P., Mooresville Mills, 476 South Main St., Mooresville, N. C. 28115

Barnes, William J., U. S. Army Natick Laboratories, Natick, Mass. 01760.

Barone, Vito S., Plastics Div., Allied Chem. Corp., 1151 Chattahoochee Ave. N.W., Atlanta, Ga. 30318.

Barrell, John M., Research & Product Dev., Mount Vernon Mills, Inc., 201 East Baltimore St., Baltimore, Md. 21202.

Beaudrot, C. Leland, Greenwood Mills, Drawer 1017, Greenwood, S.C. 29646.

Beaumont, Warren L., Proctor Chemical Co., Inc., P.O. Box 399, Salisbury, N.C. 28144.

Beck, Edward A., Technical Service Lab., A. E. Staley Manufacturing Co., P.O. Box 151, Decatur, Ill. 62525.

Bedoit, William C., Jr., Jefferson Chemical Co., Inc., P.O. Box 53300, Houston, Tex. 77052.

Belkin, Lawrence, Reeves Brothers, Inc., 1071 Ave. of the Americas, New York, N.Y. 10018.

Belser, Richard B., Georgia Inst., of Tech., Eng. Exp. Sta., 225 North Ave. N.W., Atlanta, Ga. 30332.

Belton, Ron, Hanes Dye & Finishing Co., P.O. Box 202, Winston-Salem, N.C. 27102.

Bentley, F. Edward, Jeff. Chemical Co., Inc., P.O. Box 4128, North Austin Sta., Austin, Tex. 78751.

Berard, W. Norbert, Clothing & Organic Materials Div., ARS, USDA, U.S. Army Natick Lab., Natick, Mass. 01762.

Bergman, Sylvester, Technical Serv. & Dev. Dept., The Dow Chemical Co., Abbott Rd. Bldg., Midland, Mich. 48640.

Berman, Harold W., Interchemical Corp., Berry Ave., Route 17, Carlstadt, N.J. 07072.

Bernardin, Leo J., Research & Engineering Kimberly-Clark Corp., Neenah, Wis. 54956.

Bertini, A.J., Jefferson Chemical Co., Inc., P.O. Box 4128, North Austin, Tex. 78751.

Blagman, Burton, Textile Product Dev., Research & Development Corp., Phillips-Van Heusen, 36A Industrial Way, Waldwick, N. J. 07463

Blomquist, George, Combed Yarn Div., Swift Spinning Mills, Inc., 3224 2nd Ave., Columbus, Ga. 31904.

Blumenstein, Carl R., Texize Chemicals, Inc., P.O. Box 368, Greenville, S.C. 29602.

Bolen, W. Paul, Canton Textile Mills, Inc., 50 Jeannette St., Canton, Ga. 30114.

Bond, G. Everett, Eastman Chemical Products, Inc., 3151 Maple Dr. N.E., Atlanta, Ga. 30305.

Bonge, Alfred K., Saco-Lowell Shops, Easley, S.C. 29640.

Bornet, George M., Internat. Inst. for Cotton, 10 rue du Commerce, Brussels, Belgium.

Boyle, Richard J., New Product Sales, Sales Dev., Textile Chemicals Dept., American Cyanamid Co., Bound Brook, N.J. 08805.

Breazeale, Roscoe J., Sun Chemical Corp., P.O. Box 70, Chester, S.C. 29706.

Brewer, Walter E., Textile Chemicals Dept., Nopco Chemical Co., P.O. Box 20, Newark, N.J. 07101.

Brotherton, T.K., Union Carbide Corp., Technical Center, P.O. Box 8361, South Charleston, W. Va. 25303.

Bryant, Harold W., Battelle Memorial Institute, 505 King Ave., Columbus, Ohio. 43201.

Buchholz, Siegfried, Tech. Relations Dept., BASF Colors & Chemicals, Inc., P.O. Box 8467, Charlotte, N.C. 28208.

Buck, George S., Jr., National Cotton Council of America, P.O. Box 12285, Memphis, Tenn. 38112.

Burkitt, Frank, International Institute for Cotton, c/o Shirley Institute, Didsbury--Manchester, England.

Burns, Cecil, Lyman Printing & Finishing Co., Lowenstein Research Center, Lyman, S.C. 29365

Chase, V. Lindsay, J. P. Stevens & Co., Inc., Central Research Laboratory, 141 Lanza Ave., Garfield, N. J. 07026.

Cheatham, R. J., 4619 Mandeville St., New Orleans, La. 70122.

Cicione, Robert J., Gagliardi Research Corp., 5454 Post Rd., East Greenwich, R. I. 02818.

Claiborne, J. Lyle, Technical Dev. Dept., Dixie Yarns, Inc., P. O. Box 751, Chattanooga, Tenn. 37401.

Clark, Elwood, Agrl. Stabiliz. & Conserv. Serv., U. S. Dept. Agr., 120 Marais St., New Orleans, La. 70112.

Clark, John E., Standard Chemical Products, Inc., P. O. Box 10622, Charlotte, N. C. 28201.

Clifford, Alfred T., Research & Dev. Div., Riegel Textile Corp., Ware Shoals, S. C. 29692.

Cline, Edward, Textile Fibers Pioneering Research Div., E. I. DuPont Expt. Sta., Wilmington, Del. 19898.

Coffin, Lawrence G., Fabric Research Laboratories, Inc., Dedham, Mass. 02026.

Cohen, Avner, Fireside Fabric (Canada), Ltd., 300 Sauve West, Montreal, Quebec, Canada.

Cohen, Sidney, Textile Research, Millmaster Onyx Corp., 624 Schuyler Ave., Lyndhurst, N. J. 07071.

Cole, Donald W., Cone Mills Corp., 1106 Maple St., Greensboro, N. C. 27405.

Coles, J. Gordon, Eagle & Phenix Finishing Plant, Reeves Bros., Inc., 1071 Sixth Ave., New York, N. Y. 10018.

Dabrowski, Jan, United Merchants & Manufacturers, Inc., 905 Fairfield Ave., North Augusta, S. C. 29841.

Dantzler, H. L., Kimberly-Clark Corp., Berkely Mills, Balfour, N. C. 28706.

Dean, Charles A., Russell Mills, Inc., Alexander City, Ala. 35010.

Deaton, Donald B., Chem. Res., Lyman Printing & Finishing Co., Lowenstein Research Center, Lyman, S. C. 29365.

DeLoach, James R., Graniteville Co., Graniteville, S. C. 29829.

Doering, August P., Textile Product Dev., National Starch & Chemical Co., 1700 West Front St., Plainfield, N. J. 07063.

Dreby, Edwin C., III, Scholler Bros., Inc., Collins & Westmoreland Sts., Philadelphia, Pa. 19134.

Duggan, Don C., Mission Valley Mills, P. O. Box 141, New Braunfels, Tex. 78130.

Dunham, L. A., Celanese Resins, P. O. Box 8248, Louisville, Ky. 40208.

East, Harry D., L. T. Barringer & Co., P. O. Box 87, Memphis, Tenn. 38101.

Efland, Thomas D., School Indus. Mangt., & Textile Sci., Clemson Univ., Clemson, S. C. 29632.

Elting, John P., Textile Div., The Kendall Co., P. O. Box 1828, Charlotte, N. C. 28201.

Endler, Abraham S., Apex Chemical Co., Inc., 200 South First St., Elizabeth, N. J. 07206.

Erickson, John F., Nalco Chemical Co., 180 North Michigan Ave., Chicago, Ill. 60601.

Farber, Hugh, Chemicals Dept., The Dow Chemical Co., Building 335, Midland, Mich. 48641.

Faucette, W. A., Technical Planning, 301 Eugene St., Burlington Industries, Greensboro, N. C. 27401.

Feazel, Charles E., Physical Sci. Div., South. Res. Inst., 2000 9th Ave., Birmingham, Ala. 35205.

Finley, Etta Lucille, School of Home Economics, Louisiana State Univ., P. O. Box 18107, Univ. Sta., Baton Rouge, La. 70803.

Fish, John G., Texas Instruments, Inc., P. O. Box 5303, MS 144, Dallas, Tex. 75222.

Floyd, Gene, Greenwood Mills, Drawer 1017, Greenwood, S. C. 29646.

Fort, J. R., Celanese Resins, P. O. Box 8248, Louisville, Ky. 40208.

Fortenberry, William H., Standards & Testing Br., Cotton Div., Consumer & Marketing Serv., U. S. Dept. Agr., Room 307, Building Annex, Wash., D. C. 20250.

Fortess, Fred, Celanese Fibers Marketing Co., 522 5th Ave., New York, N. Y. 10036.

Foy, Frank, Russell Mills, Inc., Alexander City, Ala. 35010.

Frump, John A., Prod. Dev. Dept., Commercial Solvents Corp., 1331 South 1st St., Terre Haute, Ind. 47808.

Gaede, A. Henry, Laurel Products Corp., 1024 N-I-85, P. O. Box 1083, Charlotte, N. C. 28201.

Getchell, Nelson F., Tech. Sect., Util. Res. Div., National Cotton Council of America, Room 502, Ring Building, 1200 18th St. N. W., Wash., D. C. 20036.

Ghionis, Constantin A., Hystron Fibers, Inc., P. O. Box 5887, Spartanburg, S. C. 29301.

Gibbon, Edmund M., Valchem, Chem. Div. of United Merchants & Mfgrs., Inc., Langley, S. C. 29834.

Gilbert, Richard D., Textile Chem. Dept., School of Textiles, North Carolina State Univ., Raleigh, N. C. 27607.

Goins, C. A., Wyandotte Chemicals Corp., 1001 Sewickley Dr., Charlotte, N. C. 28209.

Goldstein, Herman B., Chem. Products Div., Sun Chem. Corp., Wood River Junction, R. I. 02894.

Goodson, L. Aubrey, Jr., Dan River Mills, Inc., Danville, Va. 24541.

Gordon, Alex F., Old Fort Finishing Plant, Div. of United Merchants & Mfgrs., Inc., 105 2nd St., Black Mountain, N. C. 28711.

Halbedel, H. S. Harshaw Chemical Co., 1945 East 97th St., Cleveland, Ohio. 44123.

Hales, N. Battle, Commodity Office, Agr. Stabiliz. & Conserv. Serv., U. S. Dept. Agr., Wirth Building, 120 Marais St., New Orleans, La. 70112.

Hall, David M., Auburn Univ., 1327 Loftin Dr., Auburn, Ala. 36830.

Hance, L. H., Res. & Eng. Div., Fieldcrest Mills, Inc., Spray, N. C. 27352.

Handy, Carleton T., Textile Fibers Res. Lab., E. I. DuPont de Nemours & Co., Chestnut Run Location, Wilmington, Del. 19808.

Harper, Phillip V., Jr., Amines & Caustics, Jefferson Chemical Co., Inc., P. O. Box 53300, Houston, Tex. 77052.

Hart, Douglas G., Crosrol Carding Developments, Inc., Tower Dr., P. O. Box 1328, Greenville, S. C. 29602.

Hart, Lawrence, Textile Research Dept., American Enka Corp., P. O. Box 124, Enka, N. C. 28728.

Haynes, George M., Whitestone Chemical Corp., P. O. Box 2721, Spartanburg, S. C. 29302.

Heffner, Lawrence L., Cotton Chem. Finishing Lab., 212 David Clark Laboratories, Fed. Ext. Serv., School of Textiles, North Carolina State Univ., Raleigh, N. C. 27607.

Hennessey, Frederick B., Cold Spring Bleachery, P. O. Box 375, Yardley, Pa. 19068.

Herbes, William F., American Cyanamid Co., Bound Brook, N. J. 08805.

Hirata, M., Teijin, Ltd., 80 Pine St., New York, N. Y. 10005.

Hoffman, H. L., Southern Research Institute, 2000 9th Ave. South, Birmingham, Ala. 35205.

Hoiness, David E., Textile Fiber Div., E. I. DuPont de Nemours & Co., Expt. Sta., Bldg. 302, 2336 Taggart Count, Wynnwood--Wilmington, Del. 19803.

Holder, Shelby, Jr., U.S. Dept. Agr., P.O. Box 127, Stoneville, Miss. 38776.

Hollies, Norman R.S., Harris Res. Lab., 6220 Kansas Ave. N.E., Wash., D.C. 20011.

Hopper, Theodore R., Applications Tech. Serv. Lab., Organic Chem. Div., FMC Corp., P.O. Box 1616, Baltimore, Md. 21203.

Hopton, Alfred W., Market Research, Hooker Chemical Corp., 1200 47th St., Niagara Falls, N.Y. 14304.

Horvath, Steven M., Wyandotte Chem. Corp., 1609 Biddle Ave., Wyandotte, Mich. 48192.

Howard, James, The Kendall Co., P.O. Box 1828, Charlotte, N.C. 28201.

Hunter, Robert T., Product Evaluation, Colgate Palmolive Co., 105 Hudson St., Jersey City, N.J. 07302.

Hurd, Ira S., Moretex Chem. Prods., Inc., P.O. Box 2528, Spartanburg, S.C. 29302.

Ingram, Peter, Research Triangle Institute, Research Triangle Park, N.C. 27709.

Johnson, David, I.C.I. (New York), Inc.. 444 Madison Ave., New York, N.Y. 10022.

Johnson, W.W., Carolina Machinery Co., P.O. Box 22, Statham, Ga. 30666.

Jonaitis, Charles W., Commerical Dev. Dept., Chemical Div., General Mills, Inc., South Kensington Rd., Kankakee, Ill. 60901.

Jones, Frank A., Chicopee Manufacturing Co., Milltown, N.J. 08850.

Jung, S. Lup, E.I. DuPont de Nemours & Co., 1508 Emory Rd., Wlimington, Del. 19803.

Justice, G.H. Plastics Div., Allied Chemical Corp., 1 River Rd., Edgewater, N.J. 07020.

Keeton, H.M., Textile Div., Merchandise Dev. & Testing Lab., Dept. 817, Sears, Roebuck & Co., 925 South Homan Ave., Chicago, Ill. 60607.

Kenyon, Keith, Sterling Pile Fabrics Corp., 168 Stevens St., Fall River, Mass. 02722.

Kinny, Robert D., Celanese Resins, Div. of Celanese Coatings Co., 1481 South 11th St., P.O. Box 8248, Louisville, Ky. 40208.

Klein, Elias, Phys. Chem., Gulf South Research Institute, P.O. Box 26500, New Orleans, La. 70126.

Knight, Dick, Eagle & Phenix Finishing Plant, Reeves Brothers, Inc., 1071 Ave. of the Americas, New York, N.Y. 10018.

Knight, Lillian, The Singer Co., 400 East Main St., Denville, N.J. 17834.

Kosenkranius, Hans, E.I. DuPont de Nemours & Co., Wilmington, Del. 19803.

Kuzmak, J.M., American Viscose Div., FMC Corp., Marcus Hook, Pa. 19061.

Lamprinakos, John W., General Aniline & Film Corp., 140 West 51st St., New York, N.Y. 10020.

Lawrence, Hazel (Mrs.), Celanese Research Co., Summit, N.J. 07901.

Leavitt, Herbert J., Spec. Projects, Koratron Co., 617 Mission St., San Francisco, Calif. 94105.

LeBlanc, R. Bruce, Market Dev. for Textile Chemicals, Archer-Daniels-Midland Co., (ADM Chemicals), P.O. Box 2121, Charlotte, N.C. 28201.

LeClercq, George M., Organic Chem. Dept., Res. & Dev. Div., Jackson Lab., DuPont Co., P.O. Box 525, Wilmington, Del. 19899.

Lock, Michael V., Deering Milliken Research Corp., P.O. Box 1927, Spartanburg, S.C. 29301.

Looney, Franklin S., Textile Res. Lab., E.I. DuPont de Nemours & Co., 1007 Market St., Wilmington, Del. 19898.

Lord, Don S., Prod. Dev., Archer-Daniels-Midland Co. (ADM Chemicals), 10701 Lyndale Ave., Minneapolis, Minn. 55420.

Lourigan, George H., Chemicals Div., Union Carbide Corp., P.O. Box 65, Tarrytown, N.Y. 10591.

Love, James, Western Div. Res. Labs., The Dow Chemical Co., P.O. Box 351, Pittsburgh, Calif. 94565.

Lusskin, Robert M., Research & Engineering, Kimberly-Clark Corp., Neenah, Wisc. 54956.

Lyons, Richard J., United Merchants & Mfgrs. Inc., P.O. Box 2148, Greenville, S.C. 29602.

McCarty, James W., Textile Engineering, Georgia Institute of Technology, 225 North Ave. N.W., Atlanta, Ga. 30332.

MacIntyre, Robert G., The Arrow Co., 433 River St., Troy, N.Y. 12180.

McMackin, L.V., Springs Mills, Inc., Grace Bleachery, 102 Woodmont Dr., Lancaster, S.C. 29720.

Martin, William J., Fed. Ext. Serv., Clemson Univ., P.O. Box 147, Clemson S.C. 29631.

Martin, William R., Jr., American Association of Textile Chemists & Colorists, P.O. Box 12215, Research Triangle Park, N.C. 27709.

Mixon, Michael K., Russell Mills, Inc., Alexander City, Ala. 35010.

Morton, Glenn P., Auburn Univ., Auburn, Ala. 36830.

Morton, Sue B., School of Home Economics, Auburn Univ., Auburn, Ala. 36830.

Moss, Warren, D., Res. & Dev. Corp., Phillips-Van Heusen, 36A Industrial Way, Waldwick, N.J. 07463.

Moussalli, Francis S., Celanese Fibers Marketing Co., P.O. Box 1414, Charlotte, N.C. 28201.

Muyskens, Dirk, Res. & Dev. Div., FMC Corp., P.O. Box 8, Princeton, N.J. 08540.

Napier, Donald R., Petrochemical Research Div., Res. & Dev. Dept., Continental Oil Co., P.O. Drawer 1267, Ponca City, Okla. 74602.

Newton, William, Allied Chem. Corp., Baton Rouge, La. 70805.

Nickles, Bradley H., Callaway Mills Co., 409 Springdale Dr., LaGrange, Ga. 30240.

Nirenberg, Robert P., Res. & Dev. Div., DHJ Industries, Inc., 16 East 34th St., New York, N.Y. 10016.

Norwick, Braham, Beaunit Textiles, 261 Madison Ave., New York, N.Y. 10016.

Nuessle, A.C., Research Dept., Rohm & Haas Co., 5000 Richmond St., Philadelphia, Pa. 19137.

Painter, E.V., Johnson & Johnson Research Center, New Brunswick, N.J. 08903.

Patterson, R.E., College of Agriculture, Texas A&M Univ., College Station, Tex. 77843.

Patton, E.L., P.O. Box 7091, Jacksonville, Fla. 32210.

Pearce, John W., United Merchants & Manufacturers, Inc., United Merchants Research Center, P.O. Box 64, Langley, S.C. 29834.

Perkins, Henry H., Jr., Market Quality Res. Div., U.S. Dept. Agr., P.O. Box 792, Clemson, S.C. 29631.

Perry, Ronald, I.C.I. (Organics), Inc., Main St., Dighton, Mass. 02715.

Persom, Francis, Market Development, A.E. Staley Mfgr. Co., P.O. Box 151, Decatur, Ill. 62525.

Peters, Earl, Corporate Research & Technical Planning, Burlington Industries, Research Center, P.O. Box 21327, Greensboro, N.C. 27420.

Pinault, Robert W., Textile World, 252 Pleasantburg Bldg., Greenville, S. C. 29607.

Polestak, W. J., Celanese Corp., P. O. Box 1000, Summit, N. J. 07901.

Porter, John J., Textile Chemistry Dept., School of Indus. Mangt. & Textile Sci., Clemson Univ., Clemson, S. C. 29631.

Prahl, Helmut F., Dynatron Research Corp., P. O. Box 4098, Madison, Wis. 53711.

Prime, George, Laurel Products Corp., Tioga & Thompson Sts., Philadelphia, Pa. 19134.

Prince, Martin, New York Univ., 233 Fordham Landing Rd., Bronx, N. Y. 10468.

Psyras, H. G., Acetylene Chemicals Dept., General Aniline & Film Corp., 140 West 51st St., New York, N. Y. 10020.

Queen, Lawrence D., Eastman Chem. Prods., Inc., P. O. Box 531, Kingsport, Tenn. 37662.

Randall, John J., Synthron Inc., P. O. Box 307, Ashton, R. I. 02864.

Rayburn, J. A., Res. & Dev. Div., Cone Mills Corp., 1106 Maple St., Greensboro, N. C. 27405.

Roussel, John S., Louisiana State Univ., Agrl. Expt. Sta., Drawer E, Univ. Sta., Baton Rouge, La. 70803.

Runnels, Mary Maumee, Home Economics Dept., Mississippi State College for Women, Columbus, Miss. 39701.

Russell, C. F., Lynrus Finishing Co., Inc., 118 South Water St., Poughkeepsie, N. Y. 12602.

Sailer, H. R., BASF Colors & Chemicals, Inc., P. O. Box 8467, Charlotte, N. C. 28208.

Sanoh, Akira, Nichibo Co., Ltd., 200 Park Ave., New York, N. Y. 10017.

Saunders, R. H., Hercules, Inc., Wilmington, Del. 19899.

Scardiglia, Frank, Velsicol Corp., 330 East Grand Ave., Chicago, Ill. 60611.

Schmidt, Bernard G., Allied Chemical Corp., 36 Mapleleaf Lane, New Hyde Park, New York, N. Y. 11040.

Schmude, K., E. I. DuPont Co., Kingston, N. C. 28501.

Schrum, Frank, Eastman Chem. Prods., Inc., Kingsport, Tenn. 37662.

Schwinn, Allan T., Allied Chem. Corp., P. O. Box 365, Morristown, N. J. 07960.

Segall, William M., National Cotton Council of America, Room 502, Ring Building, 1200 18th St. N. W., Wash. D. C. 20036.

Seward, Kenneth A., Greenwood Mills, Inc., 111 West 40th St., New York, N. Y. 10018.

Shealy, James P., Jr., Graniteville Co., Graniteville, S. C. 29829.

Shippee, Fred B., Res. & Devlpmt. Corp., Phillips-Van Heusen, 36A Industrial Way, Waldwick, N. J. 07463.

Smith, Betty, Textiles & Clothing Dept., New York State College of Home Economics, Cornell Univ., Ithaca, N. Y. 14850.

Smith, Donald E., High Voltage Eng. Corp., South Bedford St., Burlington, Mass. 02103.

Smith, James K., Gulf South Research Institute, P. O. Box 26500, New Orleans, La. 70126.

Smith, John D., Mission Valley Mills, P. O. Box 141, New Braunfels, Tex. 78130.

Smith, Leonard, Util. Res., National Cotton Council of America, Room 502, Ring Bldg., 1200 18th St. N. W., Wash., D. C. 20036.

Smith, Louis W., Res. Dept., Dan River Mills, Inc., Danville, Va. 24541.

Smith, Rex S., New Products, Marketing Dept., Jefferson Chem. Co. Inc., P. O. Box 53300, Houston, Tex. 77052.

Smith, Wilmer, Plains Cooperative Oil Mill, P. O. Box 509, Lubbock, Tex. 79408.

Solomon, Gerald L., Tech. Serv., Valchem, Langley, S. C. 29834.

Sommer, E. Paul, American Hoechst Corp., 777 3rd Ave., New York, N. Y. 10017.

Souther, George P., The Dow Chemical Co., Building 2020, 1000 Main St., Midland, Mich. 48640.

Staley, John, 2910 Westchester Rd., Richmond, Va. 23225.

Stanley, Leonard A., Textile Div., The Kendall Co., P. O. Box 157, Bethune, S. C., 29009.

Starke, Clinton J., Organic Intermediates, FMC Corp., 633 3rd Ave., New York, N. Y. 10017.

Stevens, L. F., High Point & Chemical Corp., 601-609 Taylor St., High Point, N. C. 27260.

Stuesse, James F., Geon & Hycar Latex, B. F. Goodrich Chem. Co., 3135 Euclid Ave., Cleveland, Ohio. 44115.

Sweeney, Matthew J., Merchandise Develpmt., Design & Testing Lab., Dept. 817, Sears, Roebuck & Co., 360 West 31st St., New York, N. Y. 10001.

Tapas, John C., Velsicol Chemical Corp., 341 East Ohio, Chicago, Ill. 60611.

Tennant, Harry, Paper & Textile Div., Tee Pak, Inc., 2 North Riverside Plaza, Chicago, Ill. 60606.

Tesoro, Giuliana C., J. P. Stevens & Co., Inc., 141 Lanza Ave., Garfield, N. J. 07026.

Thornton, Raymond, I. C. I. (Organics), Inc., 55 Canal St., Providence, R. I. 02901.

Thrift, Benjamin G., Fabric Develpmt., Cone Mills Corp., 1201 Maple St., Greensboro, N. C. 27405.

Tomasino, Charles, Burlington Industries, P. O. Box B-2, Greensboro, N. C. 27402.

Towery, Jack D., Textile Res., Continental Moss-Gordin, Inc., P. O. Box 1321, Lubbock, Tex. 79408.

Troope, Walter S., Sanforized Div., Cluett, Peabody & Co., Inc., 433 River St., Troy, N. Y. 12180.

Turbak, A., Tee Pak, Inc., 915 North Michigan Ave., Danville, Ill. 61832.

Tutton, James W., Cold Spring Bleachery, P. O. Box 375, Yardley, Pa. 19068.

Wagner, George M., Hooker Chemical Corp., 1940 Ward St., Niagara Falls, N. Y. 14302.

Waldman, Milton M., McCook Res. Labs., Armour Indus. Chem. Co., 8401 West 47th St., Mc Cook, Ill. 60525.

Walker, George B., Proctor & Gamble Co., Ivorydale Tech. Ctr., Room 1-N-43, Bldg. ITC, Spring Grove Ave., Cincinnati, Ohio. 45217.

Ward, Harold G., Tech. & Textile Serv. Dept., Dyeing & Finishing Div., Avisco Div., FMC Corp., P. O. Box 455, Marcus Hook, Pa. 19061.

Wasley, William L., Fiber Chemistry Investigations, Western Util. Res. & Devlpmt. Div., U. S. Dept. of Agr., 800 Buchanan St., Albany, Calif. 94710.

Weiland, Herman G., Arkansas Co., Inc., P. O. Box 210, Newark, N. J. 07101.

Weipert, Gene, Wyandotte Chemicals Corp., 1609 Biddle Ave., Wyandotte, Mich. 48192.

West, Richard L., Organic Sect., Chem. Res. Dept., Atlas Chem. Indus. Inc., 620 Foulkstone Rd., Wilmington, Del. 19803.

Wilkerson, Vernon F., North Carolina Finishing Div., Fieldcrest Mills, Inc., Salisbury, N. C. 28144.

Willett, R. E., Quaker Chemical Corp., 3212 Flint Dr., Columbus, Ga. 31907.

Wolfgang, Ruby (Mrs.), Philadelphia College of Textiles & Science, Schoolhouse Lane & Henry Ave., Philadelphia, Pa. 19144.

Wolfgang, William G., Philadelphia College of Textiles & Science, Schoolhouse Lane & Henry Ave., Philadelphia, Pa. 19144.

Woods, John C., I. C. I. (Organics), Inc., Main St., Dighton, Mass. 02715.

Wooten, William A., Jr., L. T. Barringer & Co., P. O. Box 87, Memphis, Tenn. 38101.

Worner, Ruby K. (Dr.)., 322 North Main St., Mason City, Ill. 62664.

Wyatt, Warren F., Eagle & Phenix Div., Reeves Brothers, Inc., P. O. Box 100, Columbus, Ga. 31901.

Wyche, Murray E., News Center, Fairchild Publications, Inc., 408 Candler Bldg., Atlanta, Ga. 30303.

ATTENDANCE OF PERSONNEL FROM THE SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION FOR AT LEAST ONE SESSION OF THE CONFERENCE

Allen, Henry A. - Cotton Physical Properties Laboratory.
Andre, Jerome L., Jr. - Cotton Mechanical Laboratory.
Arthur, Jett C., Jr. - Cotton Chemical Reactions Laboratory.

Bains, Malkiat S. (Dr.) - Cotton Chemical Reactions Laboratory.
Baril, Albert, Jr. - Cotton Mechanical Laboratory.
Barker, Robert H. (Dr.) - Cotton Physical Properties Laboratory.
Benerito, Ruth R. (Dr.) (Mrs.) - Cotton Chemical Reactions Laboratory.
Beninate, John V. - Cotton Finishes Laboratory.
Berni, Ralph J. (Dr.) - Cotton Chemical Reactions Laboratory.
Bertoniere, Noelie R. (Miss) - Cotton Chemical Reactions Laboratory.
Blanchard, Eugene J. - Cotton Finishes Laboratory.
Blouin, Florine A. - Cotton Chemical Reactions Laboratory.
Bocage, Melville J. - Cotton Mechanical Laboratory
Bosworth, Louise N. (Mrs.) - Cotton Physical Properties Laboratory.
Boudreaux, Francis N. - Cotton Mechanical Laboratory.
Boudreaux, Gordon J. - Cotton Physical Properties Laboratory.
Bouquet, Norbert A., Jr. - Cotton Mechanical Laboratory
Bourdette, Vernon R. - Public Information Officer.
Boylston, Eileen K. (Mrs.) - Cotton Finishes Laboratory.
Brannan, Mary Ann F. (Mrs.) - Cotton Chemical Reactions Laboratory.
Brown, Roger S. - Cotton Mechanical Laboratory.
Bruno, Joseph S. - Cotton Finishes Laboratory.
Brysson, Ralph J. - Cotton Finishes Laboratory.

Calamari, Timothy A. (Dr.) - Cotton Finishes Laboratory.
Callegan, Arsema T. - Cotton Mechanical Laboratory.
Cannizzaro, Anna M. (Mrs.) - Cotton Physical Properties Laboratory.
Carra, Jarrell H. (Mrs.) - Cotton Physical Properties Laboratory.
Cashen, Norton A. - Cotton Finishes Laboratory.
Chance, Leon H. - Cotton Finishes Laboratory.
Cirino, Vidabelle O. (Mrs.) - Cotton Chemical Reactions Laboratory.
Conner, Charles J. - Cotton Finishes Laboratory.
Connick, William J., Jr. - Cotton Finishes Laboratory.
Conrad, Carl M. (Dr.) - Plant Fibers Pioneering Research Laboratory.
Cooper, Albert S., Jr. - Cotton Finishes Laboratory.
Copeland, Herbert R. - Cotton Mechanical Laboratory.
Creely, Joseph J. - Cotton Physical Properties Laboratory.

Daigle, Donald J. (Dr.) - Cotton Finishes Laboratory.
Daniels, Leontine Y. (Mrs.) - Cotton Physical Properties Laboratory.

Danley, Sandra R. (Mrs.) - Cotton Physical Properties Laboratory.
Danna, Gary S. - Cotton Finishes Laboratory.
deGruy, Ines, F. (Miss) - Cotton Physical Properties Laboratory.
Delmare, Ulysses J. - Plant Management.
DeLuca, Lloyd B. - Cotton Mechanical Laboratory.
Donaldson, Darrell J. (Dr.) - Cotton Finishes Laboratory.
Doyle, Bonnie P. (Miss) - Director's Office.
Drake, George L., Jr. - Cotton Finishes Laboratory.
Duffy, Charlotte P. (Mrs.) - Director's Office.

Egle, Clifton J., Jr. - Cotton Physical Properties Laboratory.
Ellzey, Samuel E., Jr. (Dr.) - Cotton Finishes Laboratory.

Fayette, Alton F. - Administrative & Plant Management.
Fiori, Louis A. - Cotton Mechanical Laboratory.
Fisher, C. H. (Dr.) - Director.
Franklin, William E. (Dr.) - Cotton Chemical Reactions Laboratory.
Frick, John G., Jr., - Cotton Finishes Laboratory.

Gallagher, Dudley M. (Dr.) - Cotton Chemical Reactions Laboratory.
Gautreaux, Gloria A. (Miss) - Cotton Finishes Laboratory.
Gentry, William T. - Engineering & Development Laboratory.
George, McLean - Cotton Physical Properties Laboratory.
Gonzales, Elwood J. (Dr.) - Cotton Chemical Reactions Laboratory.
Goynes, Wilton R., Jr. - Cotton Physical Properties Laboratory.
Guice, Wilma A. (Miss) - Cotton Finishes Laboratory.
Guthrie, John D. (Dr.) - Cotton Chemical Reactions Laboratory.

Hamalainen, Carl - Cotton Finishes Laboratory.
Habrink, Pieter - Plant Fibers Pioneering Research Laboratory.
Harper, Robert J., Jr. (Dr.) - Cotton Finishes Laboratory.
Hassenboehler, Charles B., Jr. - Cotton Physical Properties Laboratory.
Haydel, Chester H. - Engineering & Development Laboratory.
Hebert, Jacques J. - Cotton Physical Properties Laboratory.
Hennessey, Glen Rae (Mrs.) - Director's Office.
Hester, Opie C. - Economic Research Service.
Hinojosa, Oscar - Cotton Chemical Reactions Laboratory.
Hoffman, Milton J. - Cotton Finishes Laboratory.
Hoffpauir, Carroll L. - Assistant Director.
Holzenthal, Leo L. - Engineering & Development Laboratory.
Honold, Edith A. (Miss) - Cotton Physical Properties Laboratory.

Janssen, Hermann J. - Engineering & Development Laboratory.
Jones, Marie A. (Miss) - Public Information Office.
Jones, Mary Alice B. (Mrs.) - Director's Office.

Keating, Esmond J. - Engineering & Development Laboratory.
Kelly, Lauralie (Miss) - Cotton Physical Properties Laboratory.
King, Walter D. - Plant Fibers Pioneering Research Laboratory.
King, William H. - Cotton Physical Properties Laboratory.
Kingsbery, Emery C. - Cotton Mechanical Laboratory.
Knoepfler, Nestor B. - Engineering & Development Laboratory.
Koenig, Paul A. - Engineering & Development Laboratory.
Kopacz, Boleslaus M. - Assistant to the Director.
Kotter, James I. - Cotton Mechanical Laboratory.
Kullman, Russell M. H. - Cotton Finishes Laboratory.
Kyame, George J. - Cotton Mechanical Laboratory.

Lambou, Madeline G. (Mrs.) - Engineering & Development Laboratory.
Lanigan, James P., Jr. - Cotton Mechanical Laboratory.
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Tallant, John D. - Cotton Mechanical Laboratory.
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Verburg Gerald B. - Cotton Finishes Laboratory.

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Yeadon, David A. - Cotton Finishes Laboratory.
Young, Alvin H. P. (Dr.) - Cotton Finishes Laboratory.

Ziifle, Hilda M. (Miss) - Cotton Chemical Reactions Laboratory.

UNITED STATES DEPARTMENT OF AGRICULTURE
AGRICULTURAL RESEARCH SERVICE
SOUTHERN UTILIZATION RESEARCH & DEVELOPMENT DIVISION
P. O. BOX 19687
NEW ORLEANS, LOUISIANA 70119

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